1992 Annual Sampling & Analysis of Groundwater.
Reilly Tar, MN
Approved on 12-6-91

1993 Annual Sampling + Analysis of Groundwater
Reilly Ind MN
Approved 8/27/93

1994 Annual Sampling & Analysis of Groundwater
Reilly Tar, MN
Approved 4/13/94

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

FILE COPY

REPLY TO THE ATTENTION OF:

SQ-14J

MEMORANDUM

DATE: APR 13 1994

SUBJECT: Approval of the Draft PRP-Lead Quality Assurance Project Plan (QAPP) for the 1994 Annual Sampling and Analysis of Ground Water and GAC Treatment System Monitoring at Reilly Tar & Chemical Corporation Site in St.Louis Park, Minnesota

FROM: Willie H. Harris
for Regional Quality Assurance Manager

TO: John Kelly, Chief
MN/OH Remedial Response Branch

Attention: Darryl Owens, Remedial Project Manager (RPM)

I am providing approval of the Draft PRP-Lead QAPP for the 1994 Annual Sampling and Analysis of Ground Water at the Reilly Tar & Chemical Corporation Site in St.Louis Park, Minnesota. The Quality Assurance Section (QAS) received the subject QAPP on March 25, 1994 (SF QAS Log-in No.2054).

The signed approval page is attached to this memorandum. Please have the Remedial Project Manager provide final sign-off and send us a copy of the completed signature page in two weeks for our records.

Attachment

cc: K.Khanna, HSRLT-5J

QUALITY ASSURANCE PROJECT PLAN FOR SAMPLING AND ANALYSIS - GROUND WATER AND GAC TREATMENT SYSTEM MONITORING

Prepared by

The City of St. Louis Park
St. Louis Park, MN 55416

Approved by: _____ Date: 10/21/93
CARL CRAIG ~~Gary Torf~~, Quality Assurance
Director, Rocky Mountain Analytical Laboratory

Approved by: _____ Date: 10/29/93
James N. Grube, Project Manager
City of St. Louis Park, MN

Approved by: _____ Date: 4/13/94
for Quality Assurance Officer
U.S. EPA Region V

Approved by: _____ Date: _____
Remedial Project Manager
U.S. EPA Region V

Approved by: _____ Date: _____
Project Manager
Minnesota Pollution Control Agency

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:
SQ-14J

MEMORANDUM

DATE: AUG 27 1993

SUBJECT: Approval of the First *Draft* PRP-Lead Quality Assurance Project Plan for the 1993 Annual Sampling and Analysis of Groundwater at Reilly Tar & Chemical Site in St. Louis Park, Minnesota.

FROM: Curtis Ross
Acting Regional Quality Assurance Manager

TO: John Kelley, Chief
Minnesota/Ohio Remedial Response Branch

ATTENTION: Darryl Owens, Remedial Project Manager (RPM)

I am providing approval of the first *draft* PRP-Lead Quality Assurance Project Plan (QAPP) for the 1993 annual sampling and analysis of groundwater at the Reilly Tar & Chemical site in St. Louis Park, Minnesota. The QAPP package was received by the Quality Assurance Section (QAS) on August 6, 1993 (QAS Log-in SF No. 1971).

Attached to this memorandum is the signed Signature Page. Please have the RPM provide final sign-off and return a copy to the QAS for our records within two weeks.

Attachment

cc: Kaushal Khanna, HSRLT-5J

QUALITY ASSURANCE PROJECT PLAN FOR SAMPLING AND ANALYSIS - GROUND WATER AND GAC TREATMENT SYSTEM MONITORING

Prepared by

The City of St. Louis Park
St. Louis Park, MN 55416

Approved by: Lee B. Roudybush (for) Date: 10/20/92
Lee Roudybush, Quality Assurance
Director, Rocky Mountain Analytical Laboratory

Approved by: James N. Grube Date: 10/29/92
James N. Grube, Project Manager
City of St. Louis Park, MN

Approved by: Date: 8/27/93
Quality Assurance Officer
U.S. EPA Region V

Approved by: _____ Date: _____
Remedial Project Manager
U.S. EPA Region V

Approved by: _____ Date: _____
Project Manager
Minnesota Pollution Control Agency

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN STREET
CHICAGO, IL 60604

REPLY TO THE ATTENTION OF:
5Q-14J

MEMORANDUM

DATE: DEC 06 1991

SUBJECT: Approval of the Quality Assurance Project Plan for the 1992 Annual Sampling and Analysis of Groundwater at the Reilly Tar & Chemical Site in St. Louis Park, Minnesota

FROM: Valerie J. Jones
Regional Quality Assurance Manager

TO: John Kelley, Chief
Ohio/Minnesota Remedial Response Branch

ATTENTION: Darryl Owens, Remedial Project Manager

I am providing approval of the Quality Assurance Project Plan (QAPjP) for the 1992 annual sampling and analysis of groundwater at the Reilly Tar & Chemical site in St. Louis Park, Minnesota. The QAPjP package, which was received by the Quality Assurance Section (QAS) on November 13, 1991 (QAS Log-In No. 1648), consists of a revised SOP for the analysis of Low Level PAHs and changed pages to be inserted in the QAPjP of the 1991 annual sampling and analysis of groundwater.

The original signature page is attached. Please have the Remedial Project Manager provide final sign-off, and send us a copy of the completed signature page within 2 weeks of this memo.

Attachment

cc: Jodi Traub (HS-6J)
Kaushal Khanna (HSrlt-5J)

QUALITY ASSURANCE PROJECT PLAN
FOR 1992 ANNUAL SAMPLING AND ANALYSIS - GROUND WATER AND GAC TREATMENT SYSTEM MONITORING

Reilly Tar & Chemical
St. Louis Park, Minnesota

Prepared by

The City of St. Louis Park
St. Louis Park, MN 55416

Approved by: _____ **Date:** _____
Gary Torf, Quality Assurance
Director, Rocky Mountain Analytical Laboratory

Approved by: _____ **Date:** _____
James N. Grube, Project Manager
City of St. Louis Park, MN

Apporved by: Lyle C. Schmidt **Date:** 12/06/91
Valerie J. Jones, Regional Quality
Assurance Manager, U.S. EPA Region V

Approved by: _____ **Date:** _____
Remedial Project Manager
U.S. EPA Region V

Approved by: _____ **Date:** _____
Project Manager
Minnesota Pollution Control Agency

CITY OF ST. LOUIS PARK

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

October 31, 1991

Regional Administrator
United States Environmental
Protection Agency, Region 5
ATTN: Darryl Owens
Mail Code 5HS-11
230 South Dearborn Street
Chicago, Illinois 60604

Director, Solid and Hazardous
Waste Division
Minnesota Pollution Control Agency
ATTN: Site Response Section
520 Lafayette Road North
St. Paul, Minnesota 55155

President
Reilly Industries, Inc.
1510 Market Square Center
151 North Delaware
Indianapolis, Indiana 46204

RE: United States of America, et al. vs. Reilly Tar & Chemical Corporation, et al.
File No. Civ. 4-80-469

Gentlemen:

In accordance with Section 3.3 of the Remedial Action Plan for the referenced case and an Agency directive of March 11, 1991 (see attached), the City of St. Louis Park hereby submits addenda to the 1991 Sampling Plan which, when incorporated with the 1991 Sampling Plan, will result in appropriate documentation for 1992 ground water and treatment plant monitoring. To assist in your understanding of the addenda, please refer to the following comments:

Site Management Plan addendum - A new Site Management Plan is submitted. The new plan includes narrative revisions relative to Ironton-Galesville Aquifer monitoring objectives (page 3) and Drift-Platteville Aquifer monitoring (page 4); revises action dates to reflect 1992 activities; and provides submittal dates for all pages.

Quality Assurance Project Plan addendum - The accompanying page revisions reflect 1992 monitoring activities; revise narrative relative to Ironton-Galesville Aquifer monitoring objectives (page 9 of 74); and acknowledge personnel revisions in the City's project team.

5005 Minnetonka Boulevard St. Louis Park, Minnesota 55416-2290
Phone: 612-924-2500 Fax: 612-924-2663

SOP Number LM-RMA-3024 addendum - A new SOP Number LM-RMA-3024 is provided. The only difference between the new SOP (Revision 6.0) and old SOP (Revision 5.0) can be found in Section 6.1 (page 5 of 18) where paragraph one, line four includes the addition of one 4-liter continuous liquid-liquid extractor to the process.

Questions regarding document content may be addressed to this office.

Sincerely,

James N. Grube

James N. Grube
Director of Public Works

JNG/cmr
enclosure

cc: Elizabeth Thompson, Popham-Haik Law Firm (w/o enclosure)
William Gregg, ENSR Consulting & Engineering (w/enclosure)
Jerry Parr, Rocky Mountain Analytical Laboratory (w/enclosure)

R4/annsampl

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

March 11, 1991

REPLY TO ATTENTION OF: 5HS-11

CERTIFIED MAIL
RETURN RECEIPT REQUESTED
P 664 395 601 and P 664 395 602

City Manager
City of St. Louis Park
5005 Minnetonka Boulevard
St. Louis Park, Minnesota 55416

President
Reilly Industries
1510 Market Square Center
151 North Delaware Street
Indianapolis, Indiana 46204

Re: United States of America, et al vs. Reilly Tar & Chemical Corporation et al. File No. CIV4-80-469

Gentlemen:

This is to advise you that the City's 1991 Sampling Plan, including the Quality Assurance Project Plan (QAPP) for sampling and analysis of groundwater and granular activated carbon treatment monitoring has been approved. Enclosed is the signature page of the QAPP with approvals given by both the U.S. EPA Quality Assurance Officer and Remedial Project Manager and also the MPCA Project Manager. Please complete the signature page and return a copy to the Agencies as soon as possible.

It is recommended that future year submittals of the Sampling Plan be prepared as addenda to the current 1991 Sampling Plan. This would be especially useful in terms of the Quality Assurance Project Plan (QAPP) portion of the Sampling Plan. These "Addendum QAPPs" would address each of the QAPP elements but would reference the previously approved QAPP when there are no changes in the sampling procedures, analytical procedures, project organization, QA objectives, data quality objectives etc. When there are changes in the content of the QAPP elements, sampling locations, analytical parameters etc., the changes can be specified in the addendum QAPP. This would hopefully streamline the annual submittal process as well as reduce the potential for inconsistencies between the plans.

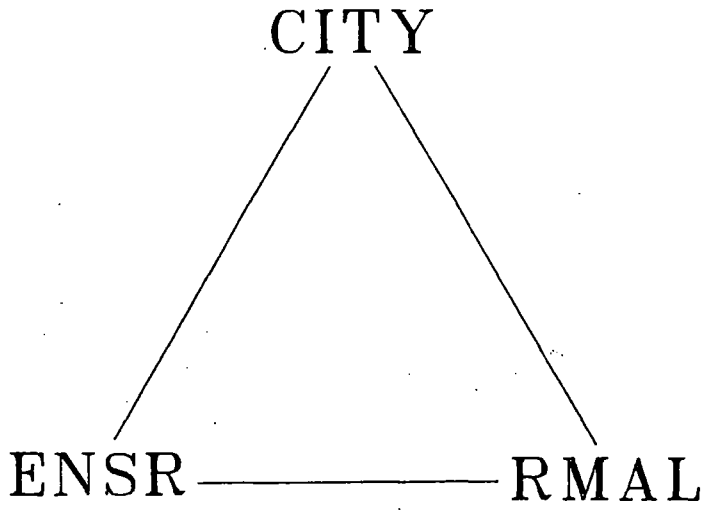
If you have any questions please contact either project leader.

Sincerely,

Darryl Owens
Remedial Project Manager
Remedial Enforcement Response Branch

Douglas Beckwith
Project Manager
Ground Water and Solid Waste Division
Minnesota Pollution Control Agency

ANNUAL SAMPLING PLAN
FOR
REILLY TAR & CHEMICAL CORP.
N.P.L. SITE
ST. LOUIS PARK, MINNESOTA



Oct., 1991

SITE MANAGEMENT PLAN

INTRODUCTION

Ground water in the City of St. Louis Park, Minnesota has been found to contain polynuclear aromatic hydrocarbons (PAH) and phenolics as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAH's in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Agency (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis Park (City), and Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into a Consent Decree, include the following concentration levels:

		Advisory Level	Drinking Water Criteria
o	Sum of benzo(a) pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
o	Carcinogenic PAH	15 ng/l	28 ng/l
o	Other PAH	175 ng/l	280 ng/l

* or the lowest concentration that can be quantified, whichever is greater

In conjunction with the implementation of remedial measures to limit the spread of PAH and phenolics, a granular activated carbon (GAC) treatment system has been installed to treat water from City wells (identified - SLP) 10 and 15. Further provisions of a Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment system and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water. The analytical data will be used to evaluate water quality by comparing the levels of PAH and/or phenolics found in the various samples with historical water quality data and with water quality criteria established in the Consent Decree-RAP. The specific objectives of the monitoring program, and therefore, the intended end use of the data vary slightly for the different aquifers being monitored in accordance with the Consent Decree-RAP.

The objective of the GAC treatment system monitoring is to assess and evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) system operations (e.g., when the carbon should be replaced), and 2) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of monitoring the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH, is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the Site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

The objective of monitoring the Ironton-Galesville Aquifer source control well W105 is to assess the levels of PAH in the aquifer. The data will be used to compare the concentration of total PAH in the samples to a threshold limit of 10 micrograms per liter of total PAH established in the Consent Decree-RAP. Also, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Water level data will be used to evaluate ground water flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to: 1) monitor the

distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground water patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells is to monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems. Ground water analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground water flow patterns in the Drift-Platteville Aquifer.

The Site Management Plan (Plan) outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, Minnesota area in accordance with the Consent Decree-RAP related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for ground water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. Although a GAC treatment system has been constructed to treat water from wells W23, W105, and the Drift-Platteville Aquifer source control wells prior to its discharge to surface water receivers, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP. Similarly, a GAC treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP.

The time period covered by this Plan is from January 1, 1994, or the date of its acceptance and approval by the Agencies whichever is later, to December 31, 1994. The next subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1994 covering the 1995 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 6.1.4, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the monitoring required under these RAP Sections has already taken place in accordance with previous Sampling Plans.

MONITORING SCHEDULE

The monitoring schedule outlined in this Plan indicates the starting criteria and the frequencies of monitoring as outlined in the RAP to determine when the GAC treatment system and wells are monitored (Tables 1 and 2). In general, the monitoring schedule will allow economies of scale in the field and in the laboratory by grouping the various monitoring events described by the RAP as much as possible. Samples will be collected within the time periods indicated on Tables 1 and 2, and all parties will be given at least 48 hours notice in advance of routine sampling.

Tables 1 and 2 summarize the GAC system/ground water monitoring schedule for the period through December 1994, and represent the minimum monitoring program that is likely to occur during the year. However, additional monitoring will take place if treated water from the GAC treatment system or ground water from active municipal drinking water wells exceeds the drinking water criteria established in the Consent Decree-RAP. This additional monitoring is described in Sections 4 and 12 of the RAP, and are reproduced in Appendix A of this Plan.

The duration of field sampling events will depend on the number and type of wells to be monitored. For estimating purposes, Drift and Platteville Aquifer monitoring wells typically are monitored at a rate of 10 wells per day, St. Peter Aquifer monitoring wells typically are monitored at a rate of 5 wells per day, and Prairie du Chien Aquifer monitoring wells typically require two to four hours or more per well to monitor.

TABLE 1

SAMPLING PLAN GAC TREATMENT SYSTEM MONITORING SCHEDULE [a]

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses[b]
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH(ppt)[c]
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625.

(a) This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1994. Sections 4 and 12 of the RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC treatment system is shown in Figure 1.

(b) Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one every 10 samples or fewer. Treated water will be duplicated at a rate of 100 percent. Feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

TABLE 2
SAMPLING PLAN GROUND WATER MONITORING SCHEDULE [a]

Source of Water	RAP Section	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
Mt. Simon-Hinckley Aquifer	5.1	SLP11,SLP12, SLP13,SLP17	Date of plan approval	Annually	PAH(ppt)[d]
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Ironton-Galesville Aquifer	6.1.4	W105 W38[e]	Date of plan approval	Annually	PAH(ppt)
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Prairie du Chien-Jordan Aquifer	7.3(A)[f]	SLP4	Start of pumping	Semi-annually	PAH(ppt) phenolics
	7.3(B)[f]	W23	Date of plan approval	Semi-annually	PAH(ppb)[g]
	7.3(C)[f]	SLP6,SLP7 or SLP9,W48	Date of plan approval	Annually	PAH(ppt)
	7.3(D)[f]	W405 or W406[h] E2,E13,H3, SLP10 or SLP15, SLP14,SLP16,W402 W403,W119	Date of plan approval	Annually	PAH(ppt)
	7.3(E)[f]	SLP5,H6,E3, E15,MTK6, W29,W40, W70,W401	Date of plan approval	Annually	PAH(ppt)
	7.3(F)	W32,SLP8, SLP10,E4, E7	Date of plan approval	Semi-annually	No Chemical analyses[i]
St. Peter Aquifer	8.1.3[j]	SLP3,W14,W24, W33,W122,W129, W133,W408,W409, W410,W411,W412, P116	Date of plan approval	Semi-annually	PAH(ppt)

TABLE 2 (continued)

Source of Water	RAP Section	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]
Drift-Platteville Aquifer	9.1.3 and 9.2.3	W420,W421, W422	Date of plan approval	Quarterly	PAH(ppb) and total phenols
	9.6	Drift:W2,W136, W15,W425,W423, W117,W116,W10, W128,W135 Platteville:W100, W101,W1,W124, W424,W121,W131, W20,W428,W19	Date of plan approval	Annually(k)	PAH(ppt) and total phenols

(a) This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1994. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one for every 10 samples or fewer, and one duplicate sample will be collected for every 10 samples.

(b) Sampling points are located on the maps shown in Figures 1 through 5. Letter prefixes to well codes are defined as follows:

W - 4-inch monitoring well

P - monitoring piezometer

SLP - St. Louis Park supply well

E - Edina supply well

H - Hopkins supply well

MTK - Minnetonka supply well

(c) Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analysis, except for those wells which prove to be inaccessible for such measurements.

TABLE 2 (continued)

(d) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

(e) Water levels in W38 will be measured each time W105 is sampled.

(f) Water levels will be measured semi-annually at these wells, except for those wells which prove to be inaccessible for such measurements.

(g) ppb = parts per billion. This signifies analysis by the Non-Criteria Method. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the Low-Level Method will be used on subsequent monitoring rounds.

(h) W405 = American Hardware Mutual, W406 = Minikahda Golf Course.

(i) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

(j) Section 8.1.3 of the Consent Decree-RAP originally specified St. Peter Aquifer monitoring requirements. Monitoring requirements for 1994, and subsequent years are now specified in the St. Peter Aquifer Record of Decision (ROD).

(k) If any of the wells listed here become damaged, destroyed or otherwise unsuitable for sampling, alternate wells will be selected by the Project Leaders for monitoring.

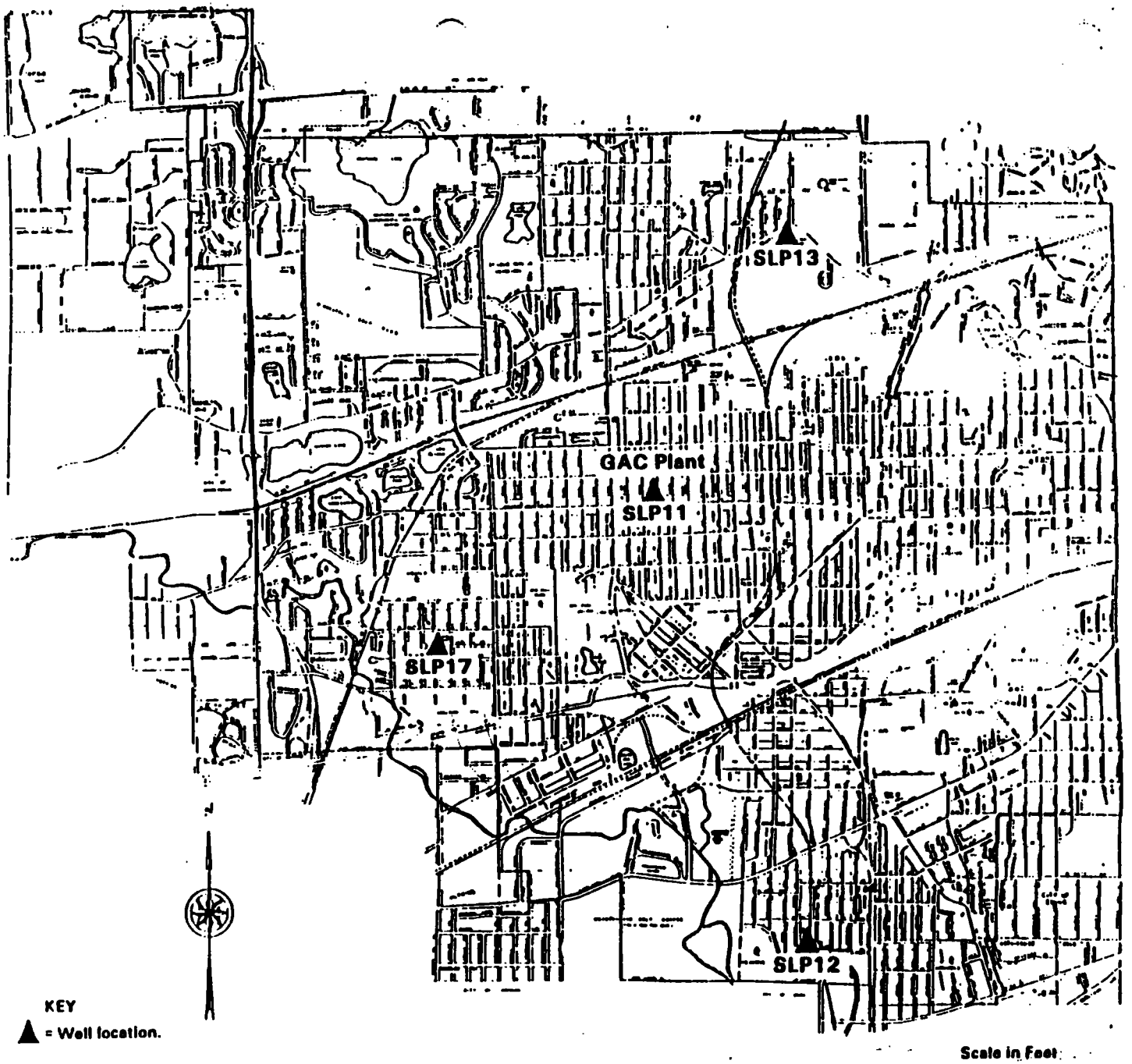


Figure 1 Location of Mt. Simon - Hinkley Monitoring Wells and St. Louis Park GAC Water-treatment Plant

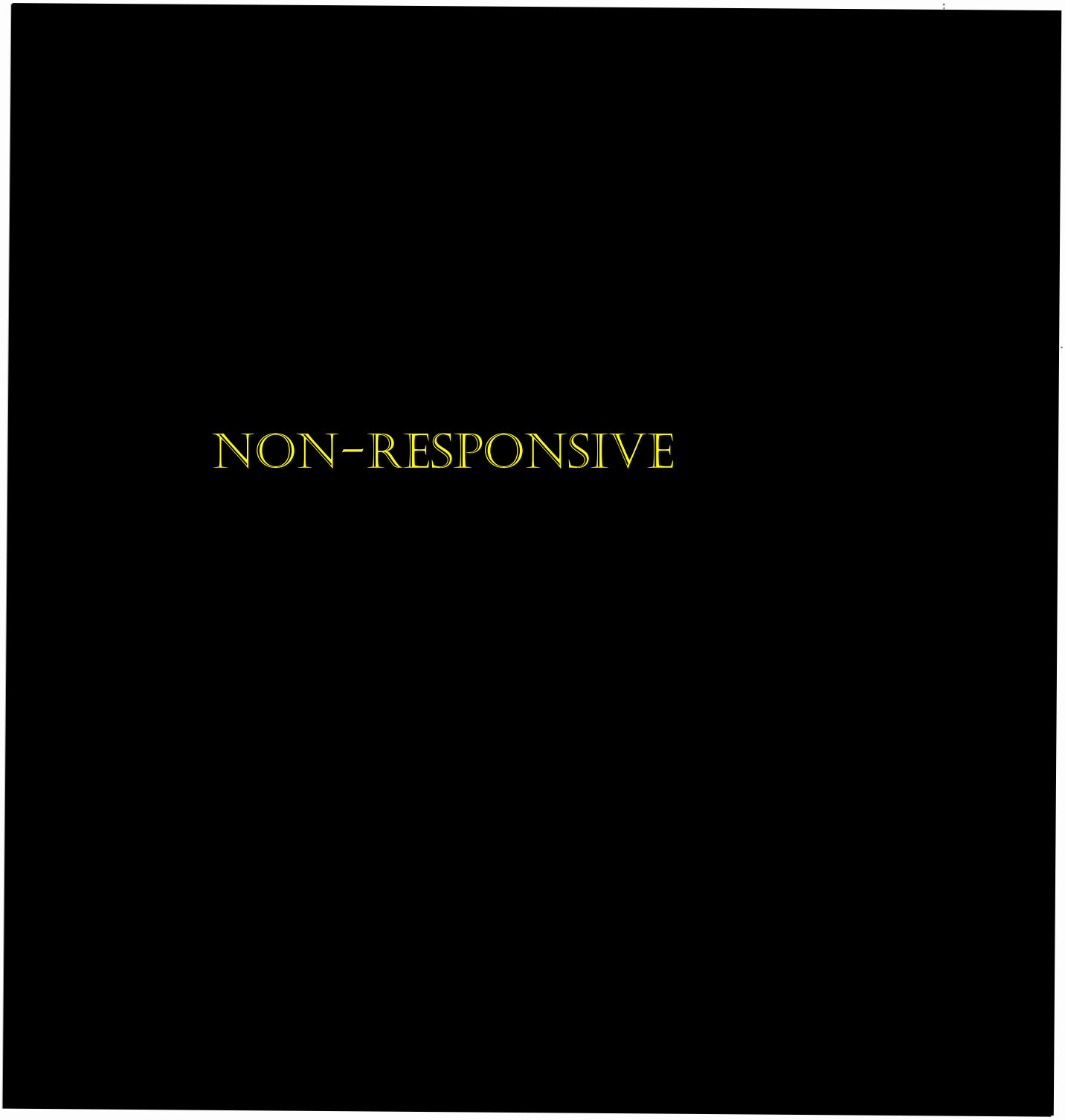


Figure 2 Location of Praire du Chien-Jordan Aquifer Wells

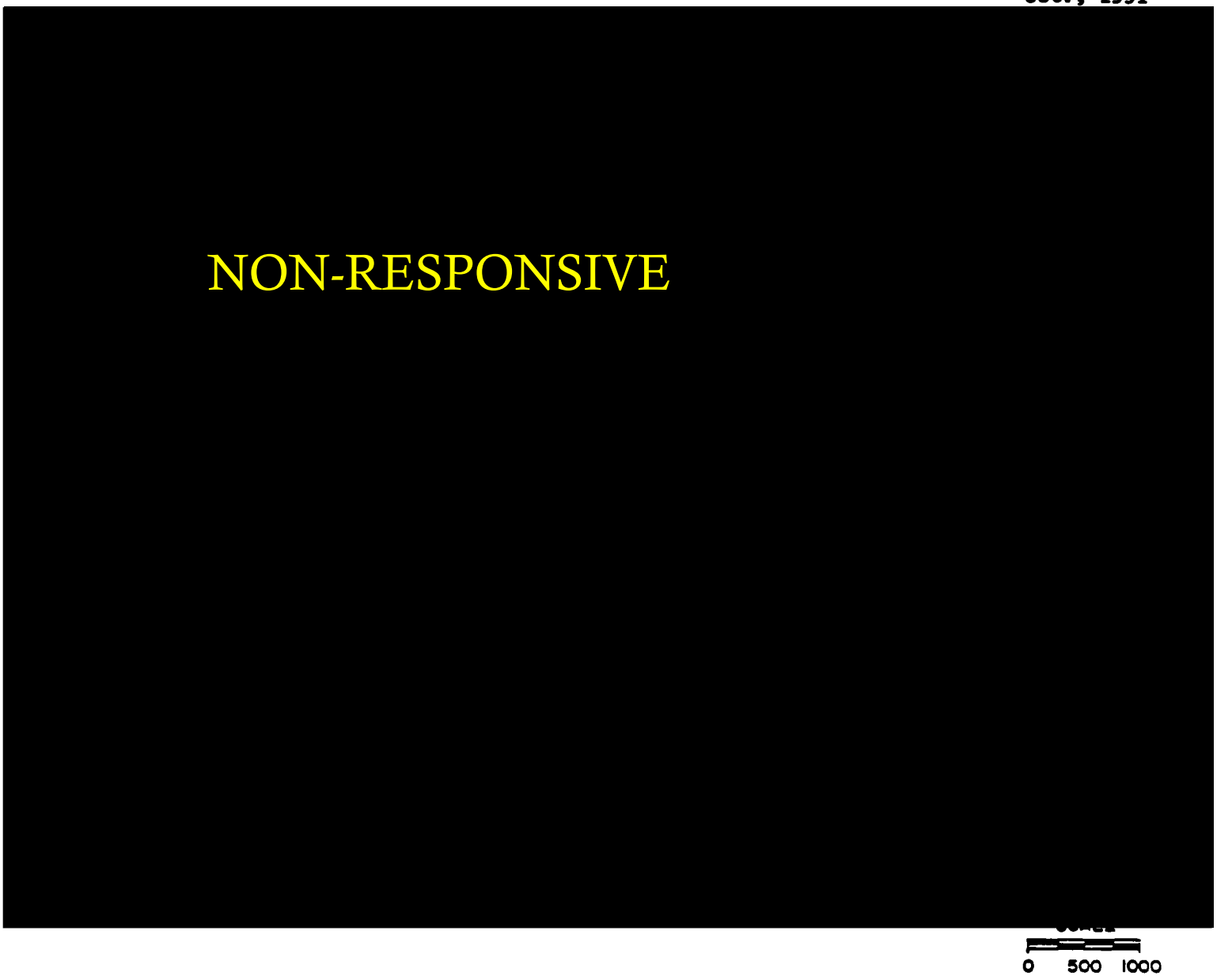


Figure 3 Location of Source and Gradient Control Wells

Reference: MGS, Miscellaneous Map Series,
M-57, Plate 1 of 2, Bedrock Geology,
by Bruce A. Bloomgren, 1985

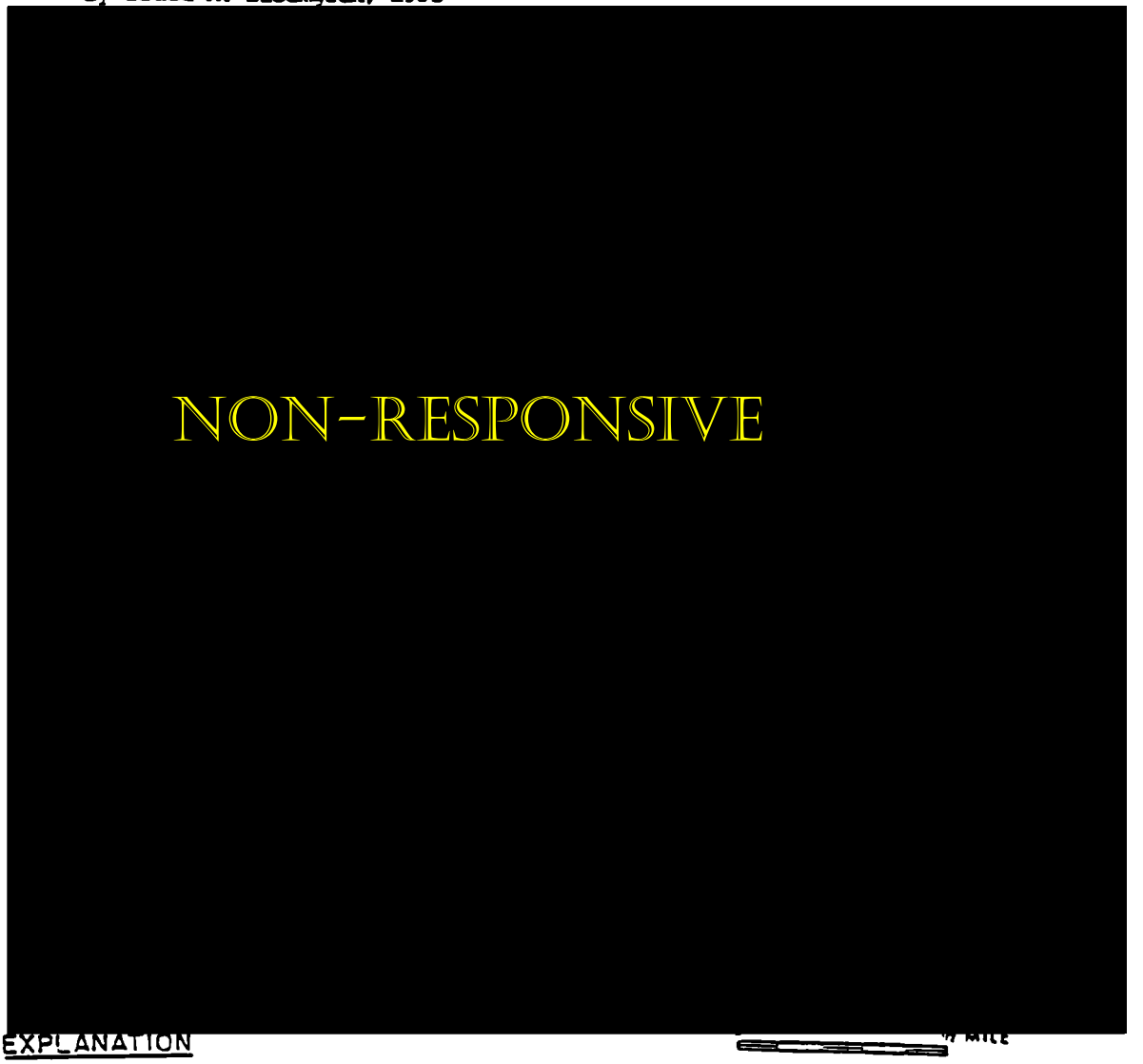


Figure 4 St. Peter Aquifer Well Locations and Bedrock Valley

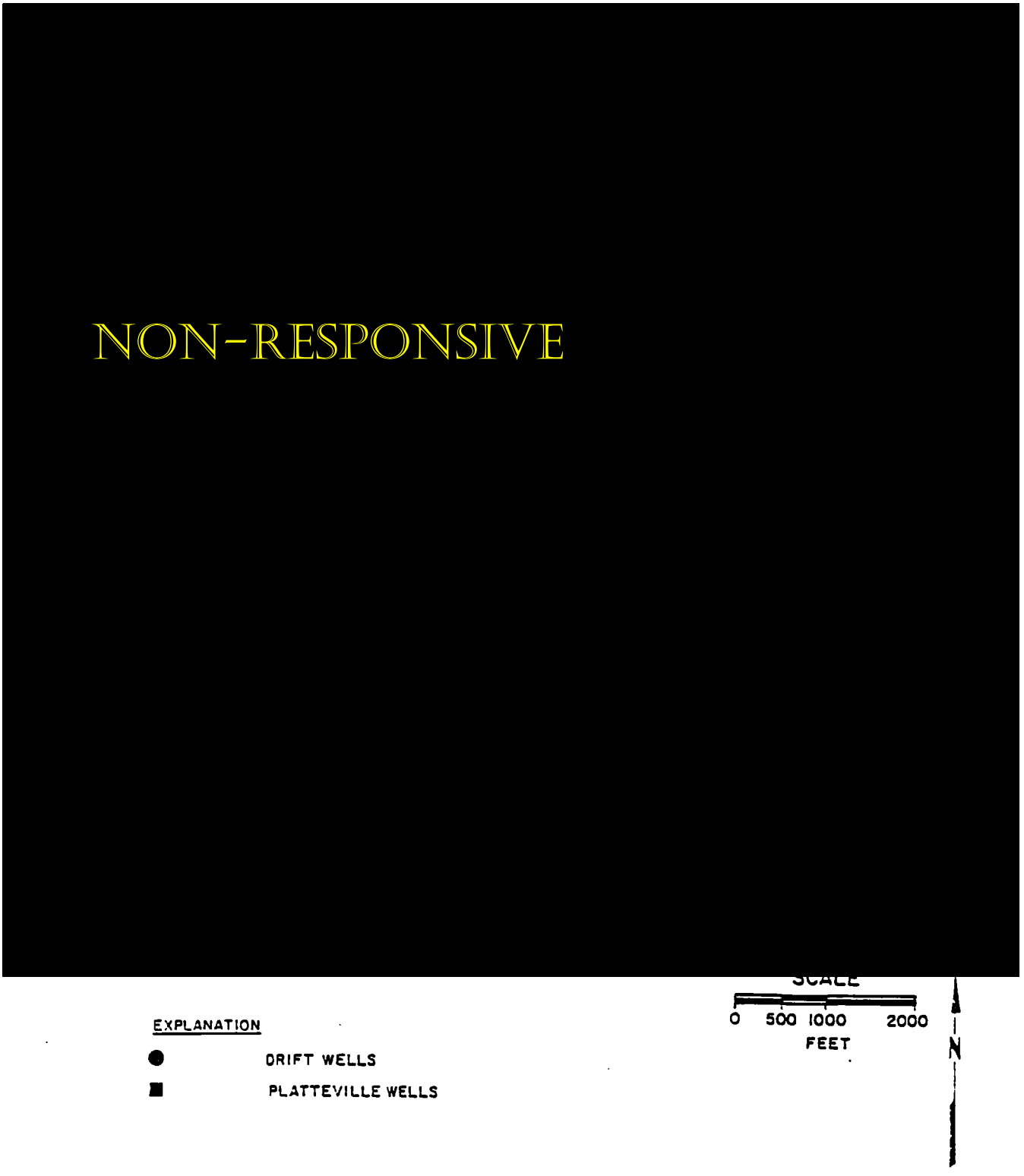


Figure 5 Location of Drift-Platteville Monitoring Wells

GROUND WATER SAMPLING PROCEDURES

An important distinction is made between the sampling procedures for active pumping wells (e.g. municipal wells) and for non-pumping monitoring wells. Active pumping wells are used on a regular basis, have dedicated pumps and associated plumbing, and have sample taps for collecting samples. Non-pumping monitoring wells may be new, or may have not been pumped for several years, and most require pumping and associated equipment for sampling. Another distinction is that the active pumping wells are typically located inside buildings whereas non-pumping monitoring wells are not.

With these considerations in mind, this Plan has been developed so that the ground water monitoring program in each aquifer meets the requirements and intent of the RAP. Ground water monitoring will be conducted in accordance with the procedures given in the Quality Assurance Project Plan (QAPP), and with "Procedures for Ground Water Monitoring: Minnesota Pollution Control Agency Guidelines", April 1985.

Water Level Measurements

Water level measurements will be made using electric tapes or weighted steel tapes. Water level measurements using steel tapes will be made by suspending a known length of tape in the well so that the bottom end of the tape is below the water level. The lower portion of tape will be coated with blue chalk that exhibits a noticeable color change when wetted. The water level measurement will be obtained by subtracting the length of wetted tape from the total length of tape suspended below the measuring point of each well.

Using the electric tape, the probe at the end of the tape will be lowered slowly in the well until contact with the water is made. Because of surface tension, readings of the water level made when the probe enters the water will differ from readings made when the probe leaves the water, thus breaking surface tension. To standardize these measurements, the second reading will always be used (i.e., the reading made when the probe leaves the water).

Water level measurement made for the purpose of defining ground water flow patterns in a particular aquifer may be performed independently from ground-water sampling, as a discrete event so as not to last more than two days. The wells will be revisited for sampling, and measurements to determine the volume of water in the well will be made at that time.

Sample Collection at Active Pumping Wells

At active pumping wells the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered. The only exception to this is the GAC treatment system monitoring under RAP Section 4.3 which includes treated water monitoring.

If it can not be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sample Collection at Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in the purging and sampling of the monitoring wells and piezometers, flexibility in the approach to sample retrieval is necessary. This Plan proposes that the sampling team be given latitude in the selection of purge/sample equipment and procedures necessary to compete the monitoring task.

Table 2 specifies the monitoring of Prairie du Chien-Jordan Aquifer monitor well W70 which is equipped with an operable dedicated submersible pump. Well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with parameter monitoring established herein.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a nondedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH, and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of

these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without prerinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

The discharge from purging monitoring wells will be handled in accordance with the Contingency Plan (Appendix B). In general, if a visible sheen can be seen on the water surface, the discharge will be routed to the sanitary sewer. Otherwise, the storm sewer or surface water discharge will be used. Non-dedicated ground water sampling or monitoring equipment that comes in contact with the ground water will be decontaminated between uses, as described in the QAPP.

ANALYTICAL PROGRAM

Tables 1 and 2 show the ground water monitoring summary as prescribed in the RAP. Indicated on the tables are the analyses required. Details of all analytical methodology can be found in the QAPP and it's appendices. All analyses will be performed at the Rocky Mountain Analytical Laboratory's (RMAL) Arvada, Colorado analytic facility. RMAL has agreed to provide a turnaround time of 30 working days from the receipt of samples to the submittal of analytical reports. The laboratory will notify the City of St. Louis Park if it can not meet this turnaround time.

Ground water monitoring will include two methods of PAH analyses depending upon the anticipated PAH concentration levels. Low-Level (nanograms per liter or part per trillion) PAH analyses will be performed utilizing selected ion monitoring (SIM) gas chromatography mass spectrometry (GC/MS). This method will be used to analyze samples from drinking water wells and from other wells for which the RAP requires drinking water criteria to be enforced (e.g., St. Peter Aquifer monitoring wells). This method is designed to analyze samples containing up to 600 nanograms per liter of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the microgram per liter range. Specific details of this methodology can be found in Appendix B of the QAPP.

Non-Criteria level (micrograms per liter or part per billion) PAH analyses, using the Scanning GC/MS Method, will be performed on samples from wells that have historically contained elevated PAH concentrations (e.g., part per million levels in well W23), and on wells that are not subject to the RAP's requirements for meeting drinking water criteria (e.g., Drift- Platteville Aquifer monitoring wells).

Two methods are required for PAH analyses because the Low-Level part per trillion SIM method is not appropriate for samples containing more than approximately 20 micrograms per liter of total PAH. Analysis of samples containing total PAH concentrations over 20 micrograms per liter, if performed with the Low-Level method, requires multiple dilutions and increases the risk of cross-contamination of the samples. This decreases the reliability of the data. Not only will multiple dilutions increase the variability of

measurements, but critical quality control information (e.g., surrogate recoveries) is lost. Therefore, for samples containing greater than 20 micrograms per liter of total PAH the analytical method that will be used is Scanning GC/MS Method as described in the QAPP.

The Scanning GC/MS Method analysis will be performed on one-liter samples, and will have detection limits of 10 micrograms per liter. For wells that are tested with this Non-Criteria method, if the analytical results of historical monitoring indicate total PAH concentrations less than 20 micrograms per liter, the Low-Level method will be used to analyze samples in 1994. This procedure will allow an evaluation of long-term PAH concentrations around the fringe PAH contamination in the Drift-Platteville Aquifer.

Depending on the circumstances and the actual PAH level, previous analytical results using the Low-Level that exceed 20,000 nanograms per liter of total PAH will indicate a switch to the Scanning GC/MS Method for 1994 sampling rounds.

REPORTING

The analytical reporting requirements of the Consent Decree and RAP are identified in Part K of the Consent Decree, and Sections 3.4, 4.3.5, 12.1.1, and 12.1.2 of the RAP. Park K requires Reilly to submit an annual progress report on March 15, 1994. This report will contain analytical reports as specified in Section 5.0 of the QAPP for this Plan, all water level measurements and chemical analyses that have not been presented in previous reports, and interpretive maps and tables, as specified in RAP Section 3.4(B) and (C). Also the effectiveness of the source and gradient control well systems in the Drift-Platteville and St. Peter Aquifers will be discussed in the annual report.

The reporting requirement for each aquifer, and for the GAC treatment system, are described below.

GAC Treatment System

RAP Section 4.3.5 requires the City to submit an annual report that presents the results of all monitoring of the GAC treatment system. Analytical results for wellhead water, feed water, and treated water will be included in this report. The report will also describe briefly the operating performance of the GAC treatment system during the previous calendar year. The GAC treatment system annual reports are due each March 15th.

Mt. Simon-Hinckley Aquifer

The monitoring data for the Mt. Simon-Hinckley Aquifer will be included in the annual report. In addition to the results of all water level measurements and chemical analyses, the report will contain a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a) pyrene and dibenz(a,h) anthracene labelled by the location of each well in accordance with RAP Section 3.4(C). Since the Mt. Simon-Hinckley Aquifer wells are monitored on an annual basis, there will be only one sampling event to report.

Ironton-Galesville Aquifer

The monitoring data for the Ironton-Galesville Aquifer will be included in the Annual Report. Since well W105 is the only well that will be sampled in this Aquifer and only one other well (W38) will be used for water level measurements, the monitoring data will be reported in tabular form as well as in map form as required by RAP Section 3.4.

Prairie du Chien-Jordan Aquifer

The monitoring data for the Prairie du Chien-Jordan Aquifer will be included in the annual report. The results of all water level measurements and chemical analyses will be included. For each of the water level measuring periods a water level contour map will be prepared with elevations labelled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well will be prepared in accordance with RAP Section 3.4(C), and a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

St. Peter Aquifer

The monitoring data for the St. Peter Aquifer will be included in the annual report. The results of chemical analyses will be reported and a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well will be prepared in accordance with RAP Section 3.4.(C). Likewise, the results of water level measurements will be provided and a water level contour map will be prepared with elevations labelled at each well in accordance with RAP Section 3.4.(B). In addition, a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

Drift-Platteville Aquifer

The monitoring data for the Drift-Platteville Aquifer including the results of all water level measurements and chemical analyses, will be presented in the Annual Progress Report. A map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h)anthracene labelled by the location of each well, and a map with phenolics concentrations labelled by the location of each well will be prepared in accordance with RAP Section 3.4. The Drift-Platteville Aquifer monitoring data will be included in the annual report to support a discussion of the results with respect to the effectiveness of the source and gradient control well systems.

QAPP/siteplan

APPENDIX A

ADDITIONAL MONITORING REQUIREMENTS

Level or Drinking Water Criterion is exceeded during the first year of operation of the system, Reilly shall immediately notify the Regional Administrator, the Director, and the Commissioner, and shall undertake such additional Monitoring as is required by Section 4.3.2.

(D) Routine Monitoring after two carbon changes shall be quarterly, unless the Regional Administrator, the Director, and the Commissioner determine that the observed service life of the carbon is too short to permit this frequency, in which case the Regional Administrator, the Director and the Commissioner shall notify Reilly of the required Monitoring frequency in accordance with Part G or H of the Consent Decree.

4.3.2. Carbon Replacement Monitoring

(A) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Drinking Water Criterion for Other PAH or exceed the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall collect two additional treated water samples at least 2 Days apart within one week of receiving the results of the exceedance sample. If the

analytical results from either one or both of the two additional samples also exceed the Drinking Water Criterion for Other PAH or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(B) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Advisory Level for Other PAH, then Monitoring of treated water shall be conducted immediately according to Section 12.1. If the results of any two samples required by Section 12.1. exceed the Drinking Water Criterion for Other PAH, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(C) If any analytical result from the additional samples taken as required by (A) or (B) above exceeds the Drinking Water Criterion for Other PAH, or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene during either

(1) within one year after the carbon treatment system is placed into service or

(2) within one year after the first carbon change if carbon was changed in the first year of operation of the carbon treatment system,

then Reilly shall conduct the Monitoring program specified in Section 4.6. Reilly shall report the results of the Section 4.6. Monitoring program to the Regional Administrator, the Director and the Commissioner within 7 Days of receiving the analytical data. If the treated water from the carbon treatment system is determined pursuant to Section 4.6. to exceed the Drinking Water Criterion for Other PAH or the Advisory Levels for Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall replace the carbon within 14 Days of making this determination. If the treated water is determined pursuant to Section 4.6. to meet the Drinking Water Criterion for Other PAH and the Advisory Levels for Carcinogenic PAH and the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then normal GAC system operation and Monitoring in accordance with Sections 4.3.1.(B) and

(C) After the first month of operation, Monitoring of feed water shall be performed quarterly until the carbon has been changed twice. If the Regional Administrator, the Director and the Commissioner determine pursuant to Section 4.3.1.(B) that the GAC system is not operating properly, Reilly may, upon receipt of such determination, be required to resume biweekly Monitoring of feed water.

(D) After two carbon changes in the GAC system, feed water shall be Monitored annually.

4.3.4. Extended Monitoring

Treated water from the GAC system shall be sampled and analyzed annually for the extended list of PAH in Part A.2. of Appendix A, using gas chromatography/mass spectroscopy (GC/MS), or other methods approved by the Regional Administrator and the Director. During this extended analysis, any compounds listed in Part A.2. of Appendix A, or any other compounds which are detected with significant peak heights that are not routinely Monitored, shall be identified and, if possible, quantified, using a mass spectral library which contains extensive spectra of PAH compounds, such as the National Bureau of Standards mass spectral library. Reilly shall analyze a sample of treated or feed water once a year for the acid fraction compounds determined by EPA Test Method 625 or by other methods approved by the Regional Administrator and the Director.

12.

CONTINGENT ACTIONS FOR MUNICIPAL
DRINKING WATER SUPPLY WELLS

12.1. Contingent Monitoring

(A) three consecutive samples yield results less than all of the Advisory Levels, in which case the sampling interval shall revert to the level specified for the affected well in Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above; or

(B) a sample yields results greater than a Drinking Water Criterion, in which case the requirements of Section 12.1.2., below, apply.

12.1.2. Exceedance of Drinking Water Criteria

(A) If the analytical result of any sample taken from an active municipal drinking water well pursuant to Section 12.1.1 exceeds the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, the Regional Administrator, the Director and the Commissioner shall be immediately notified by Reilly, and another sample shall be taken by Reilly within three Days of receiving the results of the first sample and analyzed. If the analytical result of the second sample is less than all of the Drinking Water Criteria but greater than any Advisory Level, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample and analyzed. If the results of this third sample are less than all of the Drinking Water Criteria, but greater than any Advisory Level, Reilly shall comply with the monthly sampling frequency specified in Section 12.1.1. above.

(B) If the analytical result of the second or third sample taken pursuant to Section 12.1.2.(A) above is greater than the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, Reilly shall Monitor the well weekly until such time as either: (1) three consecutive samples yield results below all of the Drinking Water Criteria, in which case Monitoring of the well shall revert to the normal schedule (including Advisory Level Monitoring as specified by Section 12.1.1. above if applicable); or, (2) three consecutive samples yield results above any Drinking Water Criterion, in which case Reilly shall immediately notify the Regional Administrator, the Director and the Commissioner. The Commissioner may then require the affected well to be taken out of service, in which case Reilly shall undertake the contingent actions specified in Section 12.2. below.

12.I.3. Analytical Turn-around Time

All Monitoring conducted pursuant to Section 12.1. shall be on a 21-Day turn-around time basis in accordance with Section 2.8.

APPENDIX B

CONTINGENCY PLAN

Contingent Actions for Contaminated Water

It is possible that groundwater contaminated with coal tar materials will be encountered during the sample retrieval operations. Groundwater generated during sample retrieval operations will be classified as contaminated if the water exhibits a discernible oil sheen or oil phase. Contaminated water will be pumped to the sanitary sewer if it contains less than ten percent organic material. Estimates of flow rate, disposal volume and water quality will be established and the Metropolitan Waste Control Commission (MWCC) will be informed before the discharge to the sanitary sewer if the estimated flow exceeds 150 gallons per workday from any individual site. Contaminated liquids containing more than ten percent organic material or failing to receive MWCC approval for discharge will be disposed of in accordance with all applicable local, state and federal rules and regulations and Part T of the Consent Decree. Uncontaminated water will be disposed of in the storm sewer or by other means acceptable to the City of St. Louis Park.

The City will be responsible for keeping the Environmental Protection Agency, Minnesota Pollution Control Agency and Reilly Tar & Chemical Corporation informed of all significant actions involving the generation of contaminated groundwater. All actions, decisions and communications by the City, Environmental Protection Agency, Minnesota Pollution Control Agency, and Reilly in dealing with contaminated soils will be in accordance with and subject to the provisions of Parts I, J, and O of the Consent Decree in the Reilly settlement.

TABLE OF CONTENTS

3. PROJECT DESCRIPTION

3.1 Background

Ground water in the City of St. Louis Park, Minnesota has been found to contain polynuclear aromatic hydrocarbons (PAH) and phenolics as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAH's in various aquifers beneath St. Louis Park and adjacent communities. Accordingly, the site of the plant operations was placed on the National Priorities List and the federal and state governments sought remediation of environmental contamination via United States District Court Case No. Civil 4-80-469.

A summary of the aquifers which underly the former wood preserving plant site, their approximate location below the surface level, the general use of the aquifers, and the relative maximum historical PAH and phenolics concentrations measured in each unit (as indicated by historical records and the federal government's Record of Decision in Case No. Civil 4-80-469) are as follows:

Aquifer	Approximate Depth (ft.)	Use	Approximate Upper Concentration of Total PAH's	Phenolics
Drift-Platteville	0 - 90	Private/Industrial/ Monitor wells	1000 ug/l offsite	10,000 ug/l offsite
St. Peter	90 - 200	Municipal/Private drinking water wells	10 ng/l offsite	16 ug/l offsite
Prairie du Chien-Jordan	250 - 500	Municipal drinking water wells	10 ug/l offsite	10 ug/l offsite
Ironton-Galesville	700 - 750	Industrial	1.4 ug/l onsite	5 ug/l offsite
Mt. Simon-Hinckley	800 - 1100	Municipal drinking water wells	16 ng/l offsite	Not detected

More extensive information relative to the identified level of PAH's in the various aquifers is provided in the following reports:

- Annual Monitoring Reports for 1988 through 1993
- St. Peter Aquifer Remedial Investigation Report (March 30, 1989)
- Drift-Platteville Aquifer (Northern Area) Remedial Investigation Report (March 30, 1989)

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis park (City), and Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into the Consent Decree - Remedial Action Plan (RAP), in the case referenced above include the following concentration levels:

		Advisory Level	Drinking Water Criteria
o	Sum of benzo(a) pyrene and dibenz(a,h) anthracene	3.0 ng/l*	5.6 ng/l
o	Carcinogenic PAH	15 ng/l	28 ng/l
o	Other PAH	175 ng/l	280 ng/l

*or the lowest concentration that can be quantified, whichever is greater

Table 3-1 lists the nominal reporting limits for the target compounds listed in the Consent Decree-RAP.

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from City wells (identified - SLP) 10 and 15. Further provisions of the RAP call for long term monitoring of the influent and effluent of the GAC treatment system and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water and compare the analytical data with water quality criteria established in the Consent Decree-RAP. The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data varies slightly for the different aquifers (Mt. Simon-Hinckley, Ironton-Galesville, Prairie du Chien-Jordan, St. Peter, and Drift-Platteville) being monitored in accordance with the Consent Decree-RAP.

3.2 Objectives and Intended Data Usage

Analytical levels for this project incorporate aspects of levels IV, and V, as defined by "Data Quality Objectives for Remedial Response Activities" (U.S. EPA, 1987). Data use categories include monitoring during implementation, site characterization, and risk assessment. It is the level of concern for low part-per-trillion concentrations of PAH that specifies a level V analytical level for this project. Level V includes non-conventional parameters, method-specific detection limits, and the modification of existing analytical methods. Rigorous QA/QC to produce data of known quality are part of this program.

TABLE 3-1
TABLE OF REPORTING LIMITS FOR TESTED PARAMETERS

CAS Number	Compound	Reporting Limit ng/L (PPT)	Reporting Limit ug/L (PPB)
271-89-6	2,3-Benzofuran	5.1	10
496-11-7	2,3-Dihydroindene	1.4	10
95-13-6	1H-Indene	0.9	10
91-20-3	Naphthalene	6.5	10
4565-32-6	Benzo(b)thiophene	0.9	10
91-22-5	Quinoline	1.4	10
120-72-9	1H-Indole	2.5	10
91-57-6	2-Methylnaphthalene	0.9	10
90-12-0	1-Methylnaphthalene	1.6	10
92-52-4	Biphenyl	4.3	10
208-96-8	Acenaphthylene	1.4	10
83-32-9	Acenaphthene	1.3	10
132-64-9	Dibenzofuran	1.0	10
86-73-7	Fluorene	1.0	10
132-65-0	Dibenzothiophene	1.1	10
85-01-8	Phenanthrene	1.3	10
120-12-7	Anthracene	1.1	10
260-94-6	Acridine	2.9	10
86-74-8	Carbazole	1.9	10
206-44-0	Fluoranthene	1.4	10
129-00-0	Pyrene	1.4	10
56-55-3	Benzo(a)anthracene	2.5	10
218-01-9	Chrysene	2.8	10
205-99-2	Benzo(b)fluoranthene	2.5	10
207-08-9	Benzo(k)fluoranthene	2.3	10
192-97-2	Benzo(e)pyrene	1.9	10
50-32-8	Benzo(a)pyrene	2.3	10
198-55-0	Perylene	2.5	10
193-39-5	Indeno(1,2,3-cd)pyrene	2.1	10
53-70-3	Dibenz(a,h)anthracene**	1.6	10
191-24-2	Benzo(g,h,i)perylene	2.8	10
205-82-3	Benzo(j)fluoranthene***	--	--
195-19-7	Benzo(c)phenanthrene*	--	--
215-58-7	Dibenz(a,c)anthracene**	1.6	--
192-65-4	Dibenzo(a,e)pyrene*	--	--
189-64-0	Dibenzo(a,h)pyrene*	--	--
189-55-9	Dibenzo(a,i)pyrene*	--	--
57-97-6	7,12-Dimethylbenz(a)anthracene	2.8	--

TABLE 3-1 (continued)

CAS Number	Compound	Reporting Limit ng/L (PPT)	Reporting Limit ug/L (PPB)
56-49-5	3-Methylcholanthrene	3.5	--
108-95-2	Phenol	--	10
95-48-7	2-Methylphenol	--	10
106-44-5	4-Methylphenol	--	10
95-57-8	2-Chlorophenol	--	10
88-75-5	2-Nitrophenol	--	10
105-67-9	2,4-Dimethylphenol	--	10
120-83-2	2,4-Dichlorophenol	--	10
59-50-7	4-Chloro-3-methylphenol	--	10
88-06-2	2,4,6-Trichlorophenol	--	10
95-95-4	2,4,5-Trichlorophenol	--	50
51-28-5	2,4-Dinitrophenol	--	50
100-02-7	4-Nitrophenol	--	50
534-52-1	4,6-Dinitro-2-methylphenol	--	50
87-86-5	Pentachlorophenol	--	50
	Total Phenolics	--	5

* Analytical standards not consistently available. It has not been demonstrated that this component can be routinely detected by this method.

** Dibenz(a,h)anthracene and Dibenz(a,c)anthracene coelute

*** Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either benzo(b)fluoranthene or benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j) fluoranthene can not be consistently separated by this method. Therefore if present, it will be detected and reported as benzo(b) and/or benzo(k)fluoranthene.

The objective of the GAC treatment system monitoring is to assess and evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) system operations (e.g., when the carbon should be replaced), and 2) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of monitoring the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH, is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the Site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

The objective of monitoring the Ironton-Galesville Aquifer source control well W105 is to assess the levels of PAH in the aquifer. The data will be used to compare the concentration of total PAH in the samples to a threshold limit of 10 micrograms per liter of total PAH established in the Consent Decree - RAP. Also, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23).

In addition to water quality data generation, water level data will be used for the purpose of determining ground water flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site.

Upon its receipt analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground water patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells is to monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems. Ground water analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground water flow patterns in the Drift-Platteville Aquifer.

In addition to the objectives for laboratory analytical data described above, field measurement data will be collected to aid in the ground water sampling procedure. In accordance with Minnesota Pollution Control Agency Guidelines (April, 1985) field measurements of temperature, pH, and specific conductance will be made for the purpose of determining that a sufficient volume of water has been purged from the well prior to sampling. The objective of those field measurements is to determine when three successive well volumes exhibiting equivalent temperature pH, and specific conductance have been purged from each monitoring well, so that representative samples may be collected.

The Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, Minnesota area in accordance with the Consent Decree-RAP related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for ground water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. Although a GAC treatment system has been constructed to treat water from well W23 and the Drift-Platteville Aquifer source control wells prior to its discharge to surface water receivers, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP. Similarly, a GAC treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP.

The time period covered by this Plan is from January 1, 1994, or the date of its acceptance and approval by the Agencies whichever is later, to December 31, 1994. The next subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1994, covering the 1995 calendar year.

4. PROJECT ORGANIZATION AND RESPONSIBILITIES

This project is being conducted in accordance with the Consent Decree-RAP for the Reilly Tar & Chemical Corporation N.P.L Site in St. Louis Park, Minnesota. The parties to the Consent Decree include Reilly, the City, EPA, MPCA, and MDH. The project organization shown in Figure 4-1 indicates the involvement of the parties to the Consent Decree, as appropriate. The City is responsible for the completion of the monitoring tasks described in this Plan. The City's Project Manager is responsible for overall project management. The City shall be assisted by two consultants in the retrieval and laboratory analysis of water samples.

ENSR Consulting and Engineering (ENSR) will be responsible for the coordination of all field sample retrieval and Enseco/Rocky Mountain Analytical Laboratory (RMAL), with analytical facilities in Arvada, Colorado, will be responsible for the coordination and completion of all laboratory analyses. Responsibilities of the key positions in the organization of RMAL are described below:

- Laboratory Project Manager: The Laboratory Project Manager is ultimately responsible for all laboratories and is the primary point of contact for issues surrounding this Quality Assurance Project Plan (QAPP), resolving technical problems, modifications to Standard Operating Procedures (SOP's) etc.

- Laboratory Project Coordinator: The Laboratory Project Coordinator is responsible for the cordination of routine day to day project activities including project initiative, status tracking, data review and requests, inquiries and general communication related to the project.

- Operations Manager: The Operations Manager is responsible for oversight of preparation and analysis of PAH samples to ensure that project objectives, requirements and Quality Assurance/Quality Control (QA/QC) criteria are met.

- Laboratory Supervisor: The Laboratory Supervisor shall be responsible for daily supervision of technicians and analysts for PAH and total phenolics analyses, including sample extraction and preparation.

- Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by the QAPP.

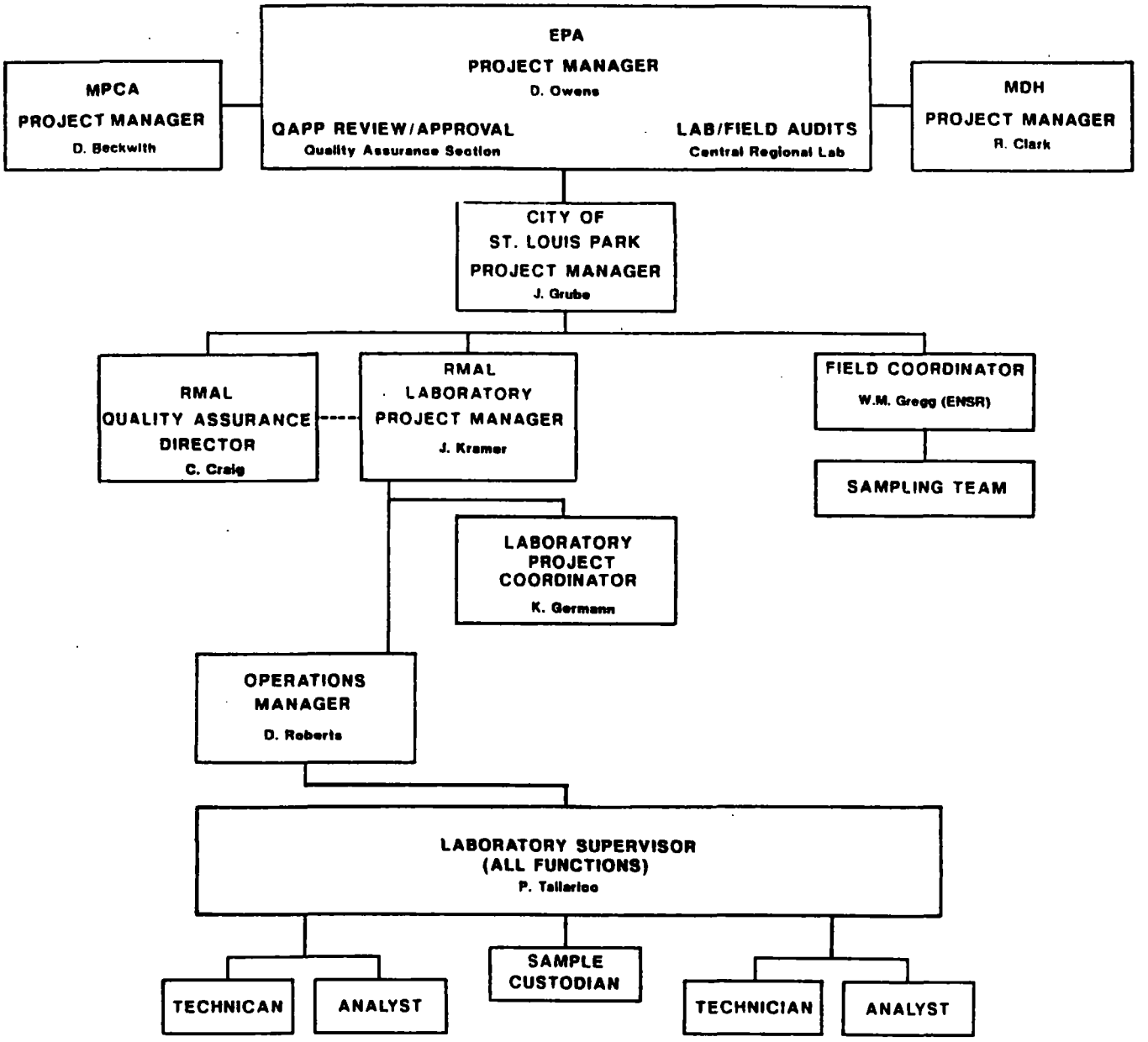


Figure 4-1 Program Organization

- Technician: The Technician is responsible for sample extraction. This requires practical experience and knowledge in the techniques of liquid - liquid solvent extraction, Kuderna - Danish evaporation, and the quantitative preparation of sample extracts for analysis.

- Quality Assurance Director: The Quality Assurance Director is responsible for overall quality control oversight, including internal audits. The Quality Assurance Director supervises an independent QA/QC department and reports directly to the Division Director and Corporate Vice President for Quality Assurance.

- Data Assessment: The evaluation of data, as it is compiled and organized in accordance with the requirements of the QAPP, is the responsibility of the Operations Manager. Additional review, evaluation, and assessment of the data is performed by the Laboratory Manager, thereby providing additional assurance that the requirements of the QAPP are met.

  The City's Project Manager shall be responsible to assess the data relative to the objectives and intended data usage identified in Section 3.2. of this QAPP.

The Sampling Team shall consist of employees of the City and ENSR. The team shall be responsible for sample collection, conducting field measurements (i.e. water level), and maintaining proper decontamination procedures stated in the QAPP.

The EPA and MPCA are responsible for review and approval of the Sampling Plan, including the QAPP. In addition, laboratory and field audits may be completed by appropriate EPA representatives. The MPCA is responsible for review of field procedures practiced by the Sampling Team. Responsibilities of the key positions in the EPA and MPCA are described below:

- EPA Project Manager: The EPA Project Manager, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA.

- EPA Quality Assurance Officer: The EPA Quality Assurance Officer, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA.

- EPA Central Regional Laboratory: The EPA Central Regional Laboratory, EPA Region 5, shall be responsible for audits of both field activities and laboratory analyses.

- MPCA Project Manager: The MPCA Project Manager shall be responsible for review and approval of the Sampling Plan, and review of field procedures practiced by the Sampling Team.

5. QUALITY ASSURANCE OBJECTIVES

The principal objectives of the QAPP pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- Completeness - a sufficient number of successful (valid) measurements to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time. For this project, the completeness objective is that 95% of the laboratory analyses and 95% of the field measurements will produce valid data. Field data will be supplemented by resampling if necessary to ensure completeness.

- Representativeness - the extent to which reported analytical results truly depict the PAH and phenolics concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.

- Accuracy and Precision - Accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.

  It is essential that representative ground water samples be retrieved for laboratory analyses. Accuracy and precision in the measurement of parameters used to monitor ground water as it is purged from monitor wells and piezometers will be achieved through the use of standard monitoring procedures carried out continuously during the sample retrieval task. Field measurement equipment will be calibrated in accordance with the manufacturer's recommendations, as outlined in Table 6-6, and appropriate corrective action will be initiated whenever measured accuracy and precision do not meet pre-established limits. Since precision and accuracy of field measurement devices are not primary objectives for the data, the quality control requirements are sufficient for the intended use of the field measurement data.

- Sensitivity - Determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of 1-5 times the instrument sensitivity, is used to determine method sensitivity (i.e. method detection limit).

- Comparability - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.

- Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

1) Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel;

2) Quality assessment through a program of regular audits and inspections to supplement continual informal review (refer to Section 12 of this QAPP);

3) Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

The St. Louis Park sampling program QAPP has been prepared in direct response to these goals. The QAPP describes the quality assurance program to be implemented and the quality control procedures to be followed by RMAL during the course of laboratory analyses in support of the various site investigation studies for the St. Louis Park Site. The Quality Assurance objectives will include field blanks, method blanks, field duplicates, surrogate spikes, matrix spikes and matrix spike duplicates. Precision, accuracy and completeness criteria are established for each parameter of interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

Objective	Frequency	Sections Discussing Criteria
Field Duplicates	10%	6.8, 11.1.4
Field Blanks	10%	6.5.2
Method Blanks	5%*	11.1.1, 15.1.3
Surrogate Spikes	100% of GC/MS analyses	11.1.2, 15.1.1
Matrix Spikes/Duplicates	5%*	11.1.3, 15.1.2

* One per group of 20 or fewer investigative samples.

6. SAMPLING PROCEDURES

Samples will be collected by ENSR and City personnel in accordance with MPCA guidelines (MPCA, 1985; Appendix A). The overall sampling program is summarized in Tables 6-1, 6-2, and 6-3, and Figures 6-1 through 6-4A. This section discusses general QAPP provisions relevant to sample collection, containerization, packaging and shipping activities (SOPs 7130 and 7510; Appendix A).

6.1 Training

All ENSR and City personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QAPP,
- Documentation requirements,
- Decontamination requirements,
- Health and Safety considerations.

Training of field personnel will be provided by the Field Coordinator or a qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed.

6.2 Document Control

Document Control for the Sampling Plan serves a two-fold purpose. It is a formal system of activities that ensures that:

1) All participants in the project are promptly informed of revisions of the QAPP; and

2) All documents generated during the course of the program are accounted for during, and at the end of the project.

This QAPP and all Standard Operating Procedure documents have the following information on each page:

- Document Number
- Page Number
- Total number of pages in document
- Revision number
- Revision date

TABLE 6-1
SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM

Sample Matrix	Field Parameter	Number of Samples	Laboratory Parameters	Number of Samples	Field Blanks	Field Duplicates	Matrix Spike[b]	Matrix Spike Duplicate[b]	Matrix Total
GAC Treated Water	X	X	PAH (ppt)	4	4	4	4	4	20
			Acid Fraction compounds[a]	1	X	1	1	1	4
GAC Feed Water	X	X	PAH (ppt)	1	X	1	1	1	4
Ground water	pH	51	PAH (ppt)	87	9	9	9	9	123
	temperature		PAH (ppb)	14	4	4	4	4	30
	Specific Conductance		Total Phenols	36	6	6	6	6	60

(a) Analysis of sample for acid fraction compounds listed in EPA Method 625 shall be in accordance with Contract Laboratory Program Statement of Work Document OLM01.0, or most recent version.

(b) Matrix spike sample/matrix spike duplicate sample shall consist of the same matrix being analyzed. Triple the normal volume when related matrix spike/matrix spike duplicate samples are to be retrieved.

TABLE 6-2
SAMPLING PLAN GAC PLANT
MONITORING SCHEDULE[a]

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses[b]
4.3.1(C)	Treated water(TRTD)	Date of plan approval	Quarterly	PAH(ppt)[c]
4.3.3(C)	Feed water(FEED)	Date of plan approval	Annually	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625.

(a) This schedule does not include certain contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1994. Sections 4 and 12 of the RAP outline the additional sampling that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC plant is shown in Figure 6-1.

(b) Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one for every 10 samples or fewer. Treated water will be duplicated at a rate of 100%. Feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

TABLE 6-3
SAMPLING PLAN GROUND WATER
MONITORING SCHEDULE [a]

Source of Water	RAP Section	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]	Duplicate Samples
Mt. Simon-Hinckley Aquifer	5.1	SLP11,SLP12, SLP13,SLP17	Date of plan approval	Annually	PAH(ppt)[d]	SLP12
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)	
Ironton-Galesville Aquifer	6.1.5	W105 W38[e]	Date of plan Approval	Annually	PAH(ppt)	W105
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)	
Prairie du Chien-Jordan Aquifer	7.3(A)[f]	SLP4	Start of pumping	Semi-annually	PAH(ppt) phenolics	SLP4
	7.3(B)[f]	W23	Date of plan approval	Semi-annually	PAH(ppb)[g]	
	7.3(C)[f]	SLP6,SLP7 or SLP9,W48	Date of plan approval	Annually	PAH(ppt)	SLP6
	7.3(D)[f]	W405 or W406[h] E2,E13,H3, SLP10 or SLP15, SLP14,SLP16,W402 W403,W119	Date of plan approval	Annually	PAH(ppt)	SLP16
	7.3(E)[f]	SLP5,H6,E3, E15,MTK6, W29,W40, W70,W401	Date of plan approval	Annually	PAH(ppt)	W70

TABLE 6-3 (Continued)

Source of Water	RAP Section	Sampling[b] Points	Start of Monitoring	Sampling Frequency	Analyses[c]	Duplicate Samples
	7.3(F)	W32, SLP8,SLP10, E4,E7	Date of plan approval	Semi-annually	No Chemical analyses[i]	
St. Peter Aquifer	8.1.3[j]	SLP3,W14,W24, W33,W122,W129, W133,W408,W409, W410,W411,W412, P116	Date of plan approval	Semi-annually	PAH(ppt)	SLP3
Drift-Platteville Aquifer	9.1.3 and 9.2.3	W420,W421, W422	Date of plan approval	Quarterly	PAH(ppb) and total phenols	W422
	9.6	Drift:W2,W136, W15,W425,W423, W117,W116,W10, W128,W135 Platteville:W100, W101,W1,W124,W424, W121,W131,W20,W428,W19	Date of plan approval	Annually (k)	PAH(ppt) and total phenols	W427,W428

(a) This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1994. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one for every 10 samples or fewer, and one duplicate sample will be collected for every 10 samples.

TABLE 6-3 (Continued)

(b) Sampling points are located on the maps shown in Figures 1 through 5. Letter prefixes to well codes are defined as follows:

W - 4-inch monitoring well
P - monitoring piezometer
SLP - St. Louis Park supply well
E - Edina supply well
H - Hopkins supply well
MTK - Minnetonka supply well

(c) Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analysis, except for those wells which prove to be inaccessible for such measurements.

(d) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

(e) Water levels in W38 will be measured each time W105 is sampled.

(f) Water levels only will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

(g) ppb = parts per billion. This signifies analysis by the Non-Criteria Method. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the Low-Level Method will be used on subsequent monitoring rounds.

(h) W405 = American Hardware Mutual, W406 = Minikahda Golf Course.

(i) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

(j) Section 8.1.3 of the Consent Decree-RAP originally specified St. Peter Aquifer monitoring requirements. Monitoring requirements for 1994 and future years are now specified in the St. Peter Aquifer Record of Decision (ROD).

(k) If any of the wells listed become damaged, destroyed, or otherwise unsuitable for sampling, alternate wells will be selected by the Project Leaders for monitoring.

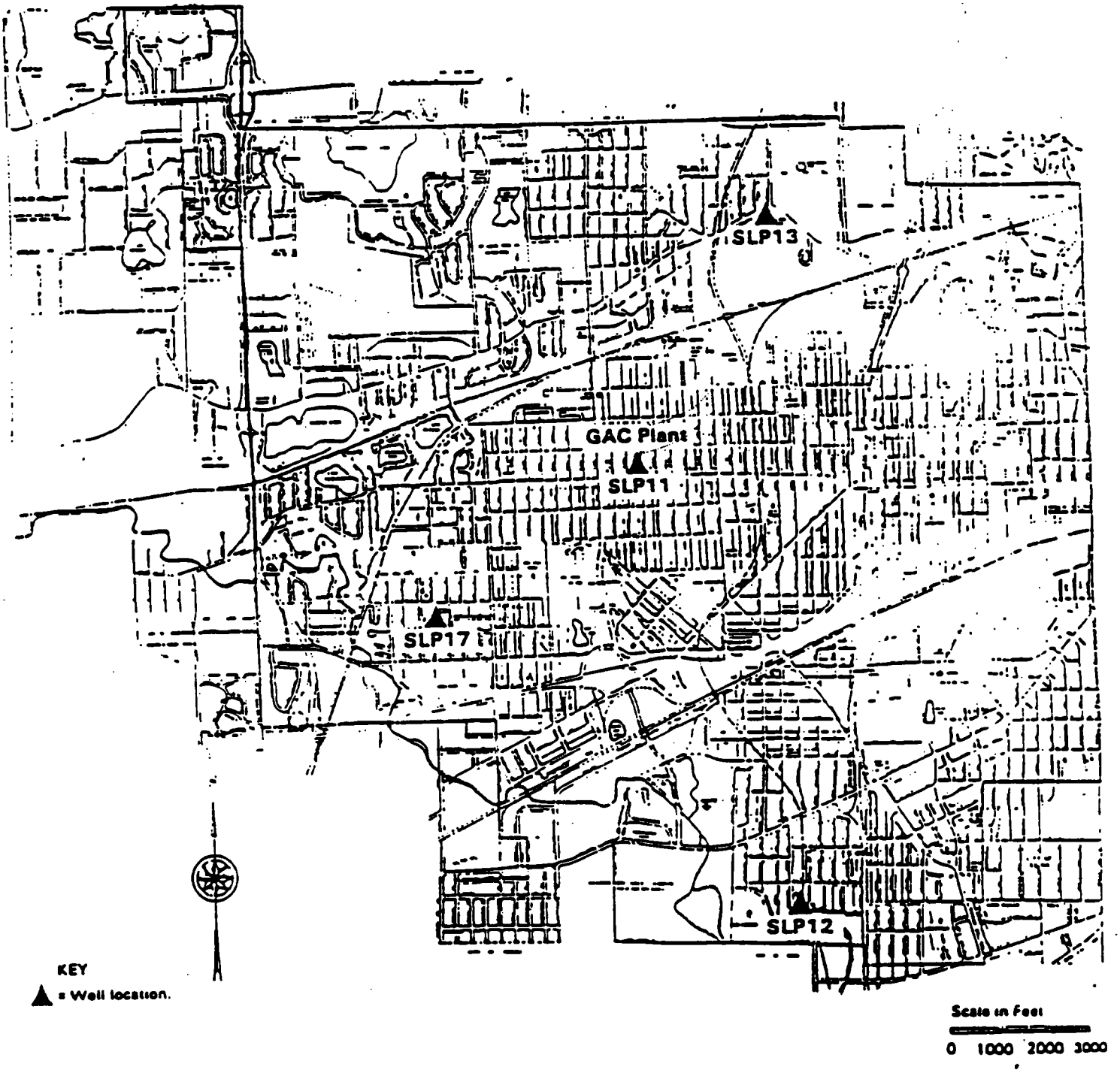


Figure 6-1 Location of Mt. Simon-Hinkley Monitoring Wells and St. Louis Park GAC Water-Treatment Plant

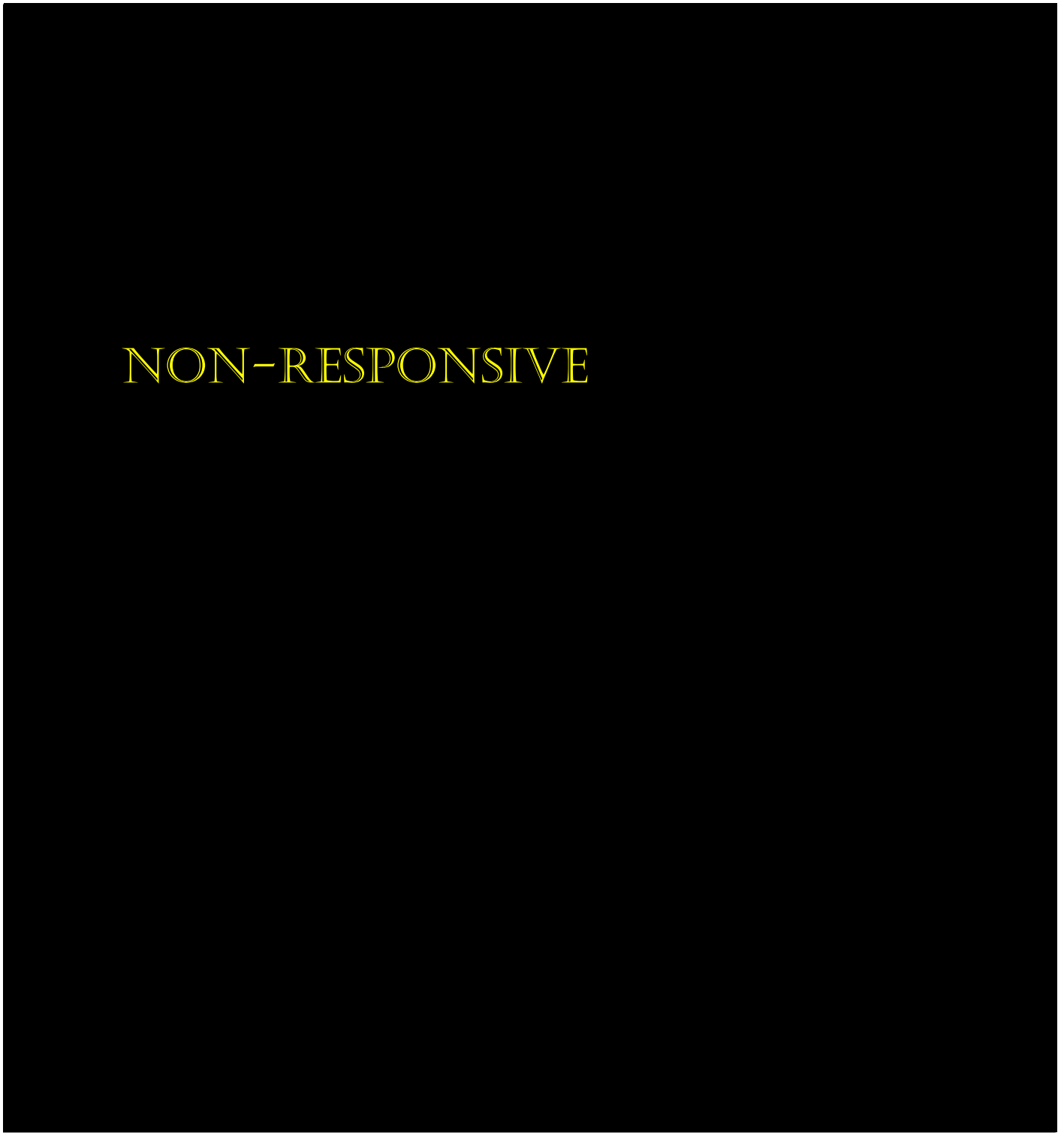


Figure 6-2 Location of Prairie du Chien-Jordan Aquifer Wells

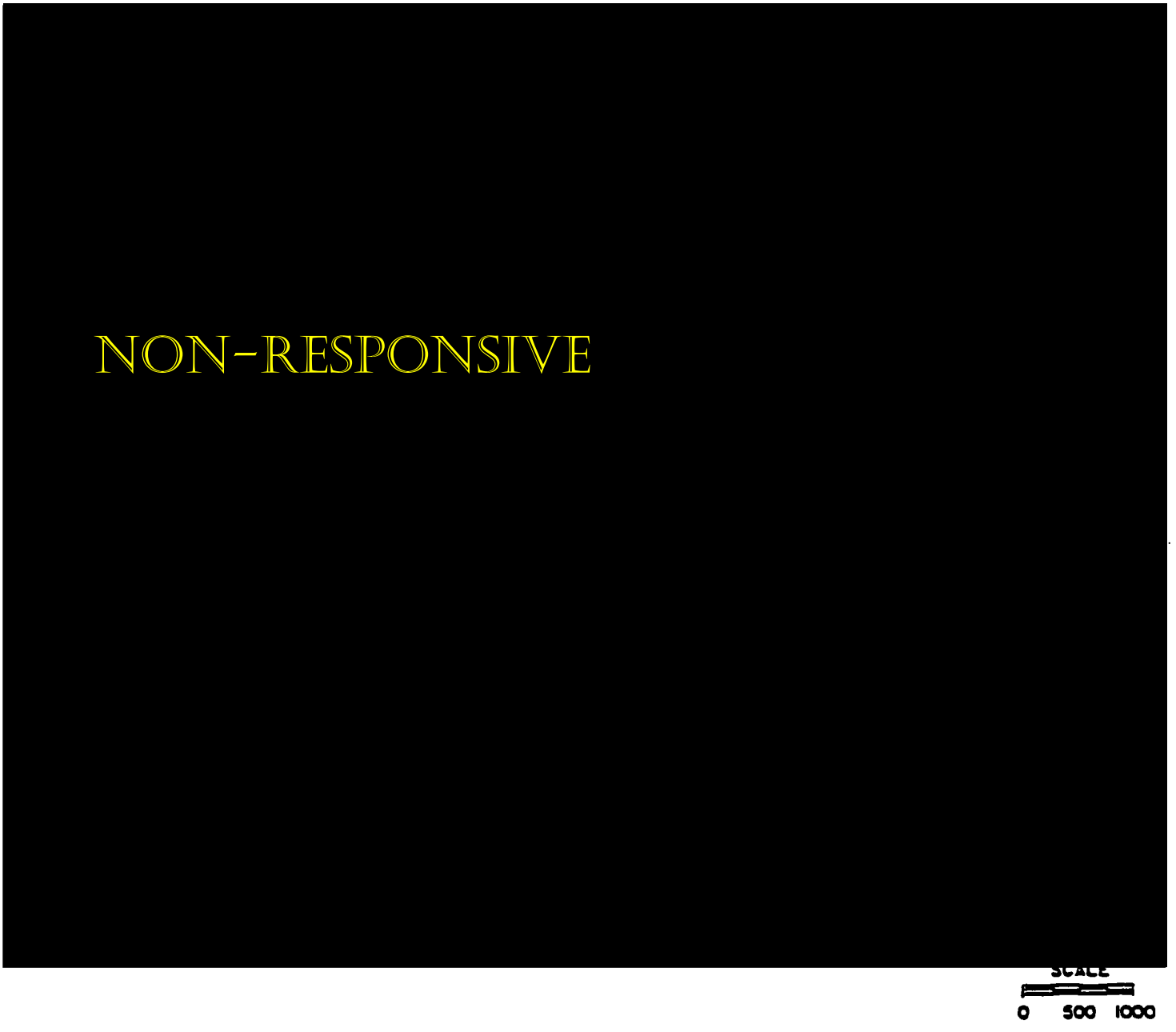


Figure 6-3 Location of Source and Gradient Control Wells

Reference: MGS, Miscellaneous Map Series,
M-57, Plate 1 of 2, Bedrock Geology,
by Bruce A. Bloomgren, 1985

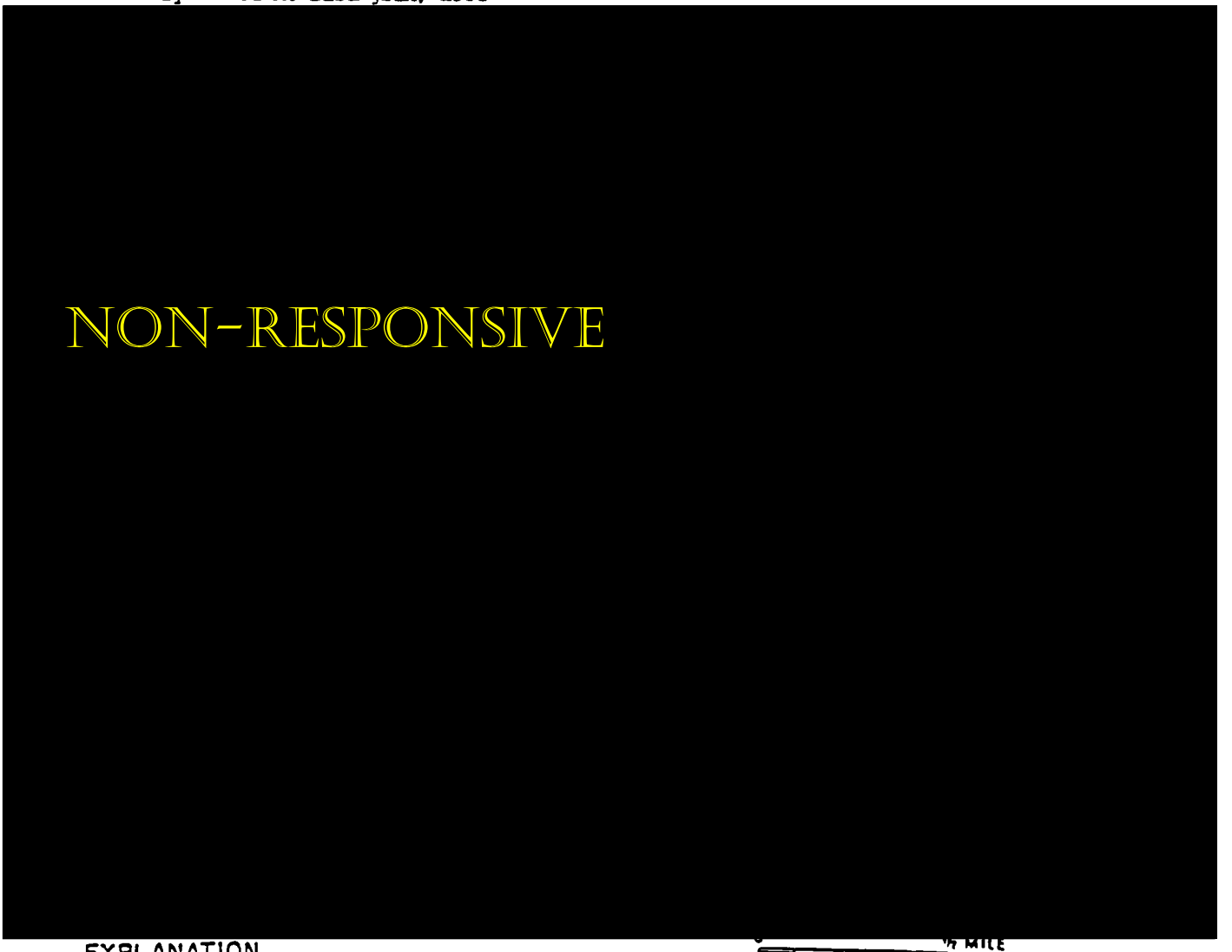


Figure 6-4 Existing St. Peter Aquifer Well Locations and Bedrock Valley

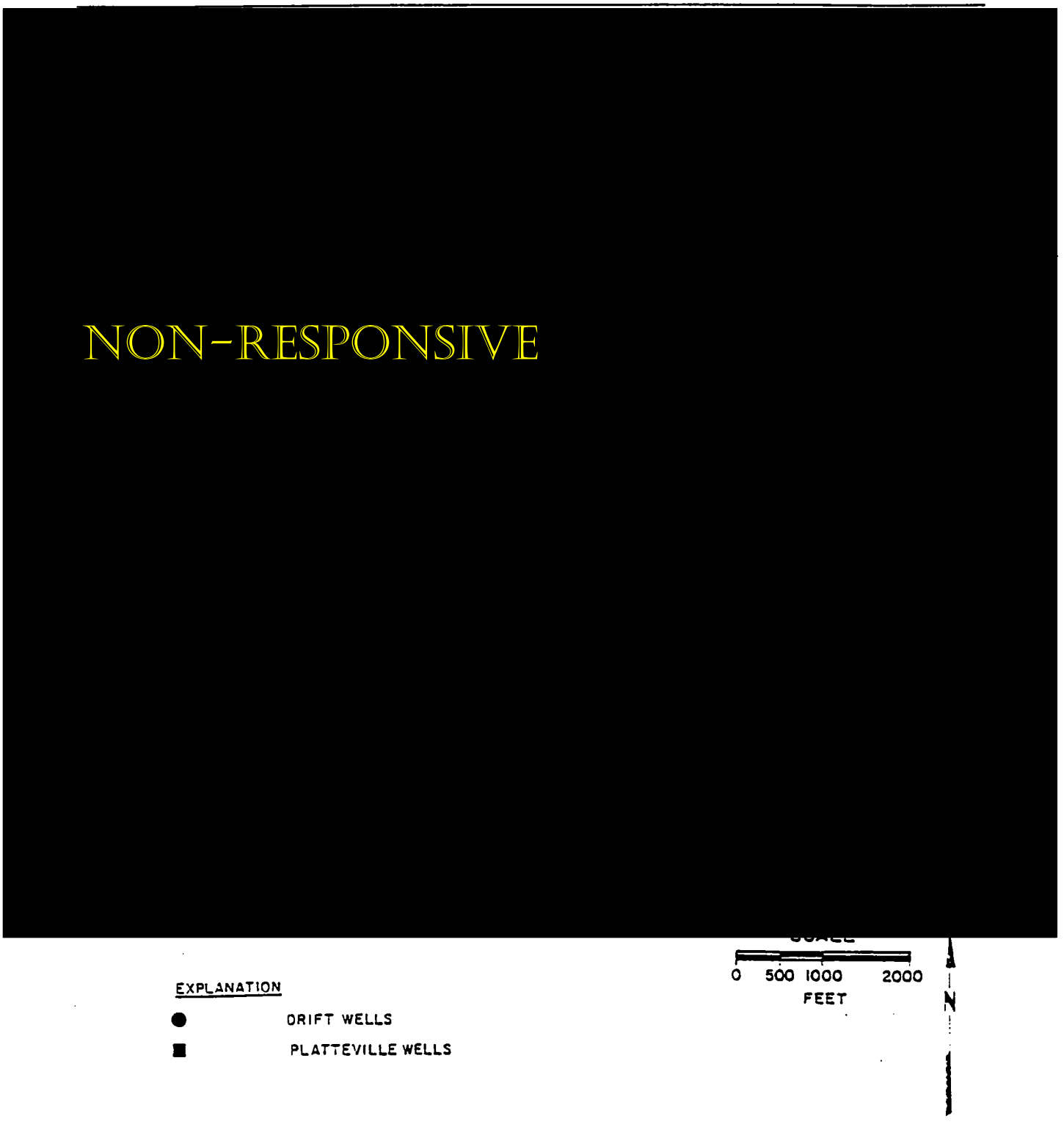


Figure 6-4A Location of Drift-Platteville Monitoring Wells

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table 6-4 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

6.3 Sample Control Procedures and Chain of Custody

In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

6.3.1 Sample Identification

Sample labels shall be completed for each sample, using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Time - A four-digit number indicating the military time of collection.

Sampler - Signature of person collecting the sample.

Remarks - Any pertinent observations or further sample description. The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

XXX-YYYYY-ZZZZZZ

TABLE 6-4
DOCUMENT CONTROL

Item	Issued By	Issued To	Archived By
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

XXX = Source Code

GAC Treatment System	= GAC
Mt. Simon-Hinckley Aquifer	= MSH
Ironton-Galesville Aquifer	= IGV
Prairie du Chien-Jordan Aquifer	= PCJ
St. Peter Aquifer	= STP
Drift-Platteville Aquifer	= DPV

YYYYY = Sampling Point Code
Well identification as abbreviated in Tables 6-2 and 6-3

ZZZZ = Date Code
Month, day, year

Those samples which will be taken in accordance with this QAPP for quality control purposes will be identified by appending to the sampling point codes the following:

Field blank = FB
Field duplicate = D
Matrix spike = MS
Matrix spike duplicate = MSD

As an example, a field blank sample taken for the Mt. Simon-Hinckley Aquifer, sampling point SLP11 on 1 January 1991 would be identified as follows:

MSH-SLP11FB-010191

During the sampling event, one sample will be taken per sampling point unless it is duplicated. Duplicate samples will be collected as specified in Tables 6-2 and 6-3. Those samples collected for matrix spike analysis will be selected at the time of sampling and labelled in the field.

Ater collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

6.3.2 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures will be followed. A sample is under custody if:

- It is in someone's possession, or
- It is in someone's view, after being in their possession, or
- It was in someone's possession and they locked it up to prevent tampering, or
- It is in a designated secure area.

Samples are accompanied by a Chain-of-Custody Record (Figure 6-5). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

Minimum information recorded on the chain-of-custody record in addition to the signatures and dates of all custodians will include:

- Sampling site indentification
- Sampling date and time
- Identification of sample collector
- Sample identification
- Sample description (type and quantity)
- Analyses to be performed.

Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. Then tear off the last copy of the form and place the original and remaining copies in the container. After the container is closed, place the custody seals on the container.

Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

6.3.3 Field Forms

In addition to sample labels and chain-of-custody forms, a bound field notebook will be maintained by the sample team leader to provide a daily record of significant events. Information to be documented in the notebook will be ground water sample collection records, calibration records, list of samples

Enseco - Rocky Mountain Analytical

4955 Yarrow Street
Arvada, Colorado 80002
303/421-6611 Facsimile: 303/431-7171

Attn: _____

CHAIN OF CUSTODY

No. 5001

Enseco Client _____

Project _____

Sampling Co. _____

Sampling Site _____

Team Leader _____

SAMPLE SAFE™ CONDITIONS

1. Packed by: _____ Seal # _____
2. Seal Intact Upon Receipt by Sampling Co.: Yes No
3. Condition of Contents: _____
4. Sealed for Shipping by: _____
5. Initial Contents Temp.: _____ °C Seal # _____
6. Sampling Status: Done Continuing Until _____
7. Seal Intact Upon Receipt by Laboratory Yes No
8. Contents Temperature Upon Receipt by Lab: _____ °C
9. Condition of Contents: _____

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by: (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: _____

Method of Shipment: _____ Airbill # _____

Received for Lab: _____ Signed. _____ Date/Time _____

Enseco Project No. _____

White and Pink Copies to Lab Yellow to Sampler

SS-001

Figure 6.5 Sample Chain of Custody Record

collected and any other pertinent information such as weather conditions, site visitors, ease/difficulty of retrieving samples, etc. All entries will be signed and dated. All members of the of the sampling team will use this notebook. The notebook will be kept as a permanent record.

6.4 Sampling Procedures - GAC Treatment System

Chain-of-custody forms will be completed and all samples shipped to RMAL's laboratory by overnight delivery on the same day they are collected.

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample and matrix spike sample will be collected in six one-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled.

The GAC treated water samples will have to be collected from two sample taps -- one for each column (see Figure 6-6). This will be done by filling three one-liter bottles from the first column sample tap and then three more bottles from the second (six from each for duplicate samples). No notations distinguishing the two taps will be made on the labels. Only four PAH bottles will be extracted and the extracts composited for analysis.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by RMAL into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Field duplicate and matrix spike duplicate samples will be obtained by filling twelve 1-liter bottles at the sampling point by the procedure described above, splitting these into two groups of six bottles, and assigning a different sample number to each of the resulting six-bottle samples. All samples will be packed, cooled to a temperature less than $4^{o}C$, and shipped on the day they are collected.

The sampling team must recognize that great care is required to collect samples for part-per-trillion-level PAH analyses that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building for at least 72 hours prior to the day on which PAH samples are to be collected. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed for at least 72 hours prior to the sampling day. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

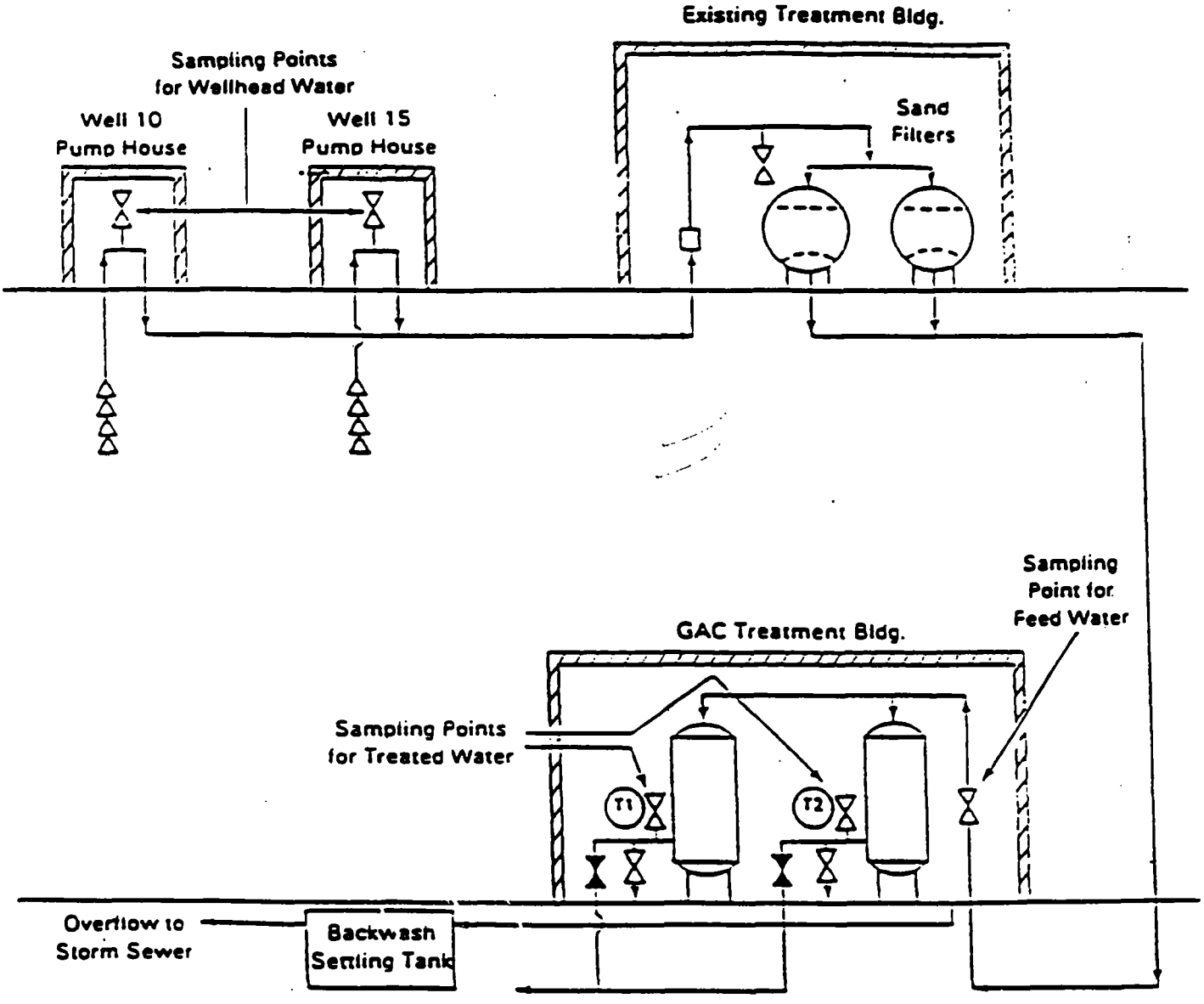


Figure 6-6 Sampling Locations

6.5 Ground Water Sampling and Water Level Measurements

Ground water samples will be collected and water levels measured in accordance with the procedures outlined in this QAPP. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells (see Table 6-3). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over-emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring points containing progressively higher concentrations of PAH or phenolics.

6.5.1 Decontamination

The field decontamination procedure to be used on sampling equipment which comes into contact with groundwater samples is as follows:

- disassemble equipment, if applicable,
- high pressure, hot water steam clean, using potable water.

The laboratory decontamination procedure to be used on sampling equipment which comes into contact with groundwater samples is as follows:

- disassemble equipment
- rinse with methanol
- scrub with hot soapy water
- rinse three times with hot deionized water
- set on aluminum foil, dull side up, air dry
- bake for one hour at 200^{o} C
- wrap with aluminum foil, dull side in

6.5.2 Field Blanks

Field blank samples will be prepared by transferring contaminant-free deionized water, provided by RMAL, into sample bottles in a fashion as closely similar to actual sample collection as possible. This will involve collecting samples through any non-dedicated sample equipment that is decontaminated between samples. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area where samples are being collected at a rate of one per day or where more than ten samples are collected in a day at a rate of one field blank per ten samples.

6.5.3 Sample Containers (See Table 6-5)

For PAH and phenolics, 1 liter amber glass bottles will be used. Caps will be fitted with pre-cleaned teflon liners. Six bottles are required for each Low-Level PAH sample collected and two bottles for each Non-Criteria PAH and Extended Analysis sample collected. One 16 ounce glass bottle with 2 milliliters of 50 percent sulfuric acid is required for total phenolics. An independent commercial firm shall provide precleaned bottles to RMAL for use on this project.

In the event RMAL is required to prepare bottles for sampling, the bottles will be prepared as follows:

1. Wash bottles with hot detergent water.

2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.

3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.

4. Allow to air dry in a contaminant free area.

5. Caps and liners must be washed and rinsed also.

   Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

6.5.4 Sample Collection - Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in purging the monitoring wells and piezometers, flexibility in the approach to the method of well purging is necessary. This QAPP proposes that the sampling team be given latitude in the selection of purge equipment necessary to complete the task (various pumping equipment and procedures that may be used for purging monitoring wells are described in SOP 7130 and MPCA's "Procedure

TABLE 6-5
SAMPLE CONTAINERS, PRESERVATION PROCEDURES, AND
MAXIMUM HOLDING TIMES

Parameter	Containers[1]	Preservation[2]	Maximum Holding Time[3]
Water:			
PAH (PPT)	Six 1-liter amber glass bottles, Teflon-lined caps	cool, to 4°C; protect from light	5 days (until extraction), 40 days after extraction
PAH (PPB)	Two 1-liter amber glass bottle, Teflon-lined caps	cool, to 4°C, protect from light	5 days (until extraction), 40 days after extraction
Phenolics (Acid Fraction)	Two 1-liter amber glass bottle,	cool, to 4°C	5 days (until extraction), 40 days after extraction
Phenolics (Total	Two 16 oz. clear glass bottle	cool, to 4°C 2 ml 50% H_2SO_4	28 days

Ref: Federal Register Guidelines/Vol.49, No.209/Friday, October 26, 1984/p. 43260.

1 Matrix spike samples shall consist of the same matrix being analyzed, therefore triple the normal volume when a related matrix spike sample and matrix spike duplicate are to be retrieved.

2 Sample preservation will be performed immediately upon sample collection.

3 Samples will be analyzed as soon as possible after validated time of sample receipt (VTSR). The times listed are the maximum times that samples may be held before analysis and still be considered valid.

for Ground Water Monitoring"; Apprendix A). In all cases where no dedicated pump exists, samples will be retrieved using laboratory - cleaned, stainless steel or teflon bailers as described below.

Table 6-3 specifies that Prairie du Chien-Jordan Aquifer monitor well W70, and St. Peter Aquifer monitor wells W24 and W33 be monitored. Each well is equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with monitoring parameters established herein. In the event the dedicated pump within any individual well is inoperable, the pump will be removed and purging/sampling procedures will be as established below.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a nondedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope. All nondedicated purging and sampling equipment will be decontaminated before use and between sampling points as described in Section 6.5.1.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without prerinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

6.5.5 Sample Collection - Pumping Wells

At active pumping wells the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered.

If it can not be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

6.6 Sample Preservation, Shipment and Storage

Packaging and shipment of samples shall be in accordance with SOP 7510 (Appendix A). The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAH's are known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples for gas chromatography mass spectrometry (GC/MS) analysis will be extracted within five days of validated time of sample receipt as per Contract Laboratory Program (CLP) Statement of Work (SOW) Document OLM01.0, or most recent version. The analysis will be completed within 40 days following extraction. The holding time for total phenolics is 28 days from sample collection to analysis.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C or less. An overnight carrier will be selected to insure delivery at the laboratory within 24-36 hours after collection.

Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C.

6.7 Field Measurement Equipment

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table 6-6 summarizes the parameters to be monitored, the instruments to be used for each measurement, procedures including calibration and frequency, and quality control criteria (also refer to Appendix A, SOP 7320, Calibration and Operation of Hydrolab Water Quality Monitor).

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel.

TABLE 6-6
FIELD MEASUREMENT EQUIPMENT QUALITY CONTROL

Device	Calibration	Routine Check Method	Routine Check Frequency	Routine Check Control Limits
pH Meter (Hydrolab)	Standardize in three or more standard buffer solutions	Calibration check-analyze standard buffer solution	After every sample	±0.1 pH units
		Analyze duplicates	After every sample	±0.1 pH units
Conductivity Meter (Hydrolab)	Standardize using two or more KCL solutions	Calibration check-analyze standard KCL solution	1/10 samples	±1% of range being used
		Analyze duplicates	1/10 samples	±1% of range being used
NBS* Thermometer	Factory calibrated	Not required	Not required	±0.1° C
Water Level Measurement Device (Electric)	Factory calibrated	Not required	Not required	±0.01 Ft.

* NBS - National Bureau of Standards

6.8 Duplicate Samples

Duplicate samples will be collected by alternately filling sample bottles from the source being sampled. For six liter sample collection one bottle will be filled for the sample, then one bottle for the duplicate, then a second bottle for the sample and then a second bottle for the duplicate, etc. Duplicates will be taken for each analysis type and each sample type, at a rate of one duplicate sample being collected for each ten samples, with a minimum of one duplicate for any sample batch. There are two sample types for this program: GAC treatment system water and ground water.

For purposes of fulfilling the 10% duplicate requirement, all the sampling points shown on Table 6-3 are the same sample type and have been chosen to maximize the frequency of sample duplication from pumping wells and monitor wells where experience indicates sampling is easiest, thereby insuring consistency of results.

7. SAMPLE CUSTODY

The St. Louis Park sampling program is a cooperative effort between the City and ENSR, whose responsibilities include sample retrieval, and RMAL, whose responsibilities include sample analysis. Proper sample handling and analysis is essential to the success of the study, therefore a formal sample custody procedure has been developed to insure the integrity of all samples. Sections 6.4 and 6.5 discuss field sampling aspects and Section 6.6 outlines procedures for sample preservation, shipment, and storage. This section covers quality related activities from receipt of samples at the RMAL analytical facilities through issuance of validated analytical data and the storage of data in the final evidence file.

7.1 Security and Recordkeeping

Samples entering the RMAL analytical facilities located in Arvada, Colorado, proceed through an orderly chain-of-custody sequence specifically designed to insure continuous integrity of both the sample and documentation.

Appendix A contains Standard Operating Procedures (SOP's) which address the following aspects of facility security and sample custody

- Building Security - SOP No. LP-RMA-0001
- Sample Log-in - LP-RMA-0003
- Use of Project Assignment Record - LP-RMA-0004
- Sample Receipt and Chain of Custody - SOP No. LP-RMA-0005

7.2 Final Evidence File

The final evidence (or data) files will be maintained for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports, raw data, continuing calibration checks, DFTPP tune, detection limits, chain of custody documentation, quality control data for blanks and matrix spikes, results forms, and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues is included in the package. The City will maintain these files in a secure, limited access area under the custody of the Project Manager. RMAL maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

8. CALIBRATION PROCEDURES

Calibration is required to ensure that field and laboratory analytical systems are operating correctly and functioning at the proper sensitivity to meet established detection limits. For this project, calibration is required for field measurements of temperature, pH, and specific conductance. Appendix A contains SOP 7320 that describes calibration procedures for field measurement instruments. This project also requires calibration for the four laboratory analyses (Low-Level, Non-Criteria, Extended, Phenolics). These four analyses are defined in Section 9 of this QAPP.

The specific calibration requirements for each of these analyses are summarized in the subsections below.

8.1 Low-Level (ppt) Analysis

The calibration requirements are described in detail in the Standard Operating Procedure for ppt PAH analyses (Appendix B). The discussion below highlights the key aspects of the calibration requirements.

Prior to use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

A midpoint calibration standard is analyzed at the start of each 12 hour calibration sequence and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound listed in Table 8-1 is calculated.

These daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within $\pm$35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within $\pm$35 percent of the corresponding calibration curve value, the system is out of control and corrective action must be performed.

The quantitation mass ion, which represents the 100% abundance ion, is selected for quantitation and for the daily response factor measurement. The second ion, or confirmation ion, is used for confirmation of the identification. The daily response factor for the quantitation mass ion is compared to the initial calibration curve. During the analysis of the daily calibration standard the percent abundance of the confirmation ion is obtained. This percent abundance is used for identification purposes for samples analyzed during that day. The percent abundance values shown in Table 8-1 are typical values.

TABLE 8-1 TARGET COMPOUNDS AND KEY IONS
FOR LOW LEVEL PAH ANALYSES

CAS NO.	COMPOUND	QUANTITATION MASS ION	CONFIRMATION ION (% ABUNDANCE)
271-89-6	2,3-Benzofuran	118	90 (52)
496-11-7	2,3-Dihydroindene	117	118 (57)
95-13-6	1H-Indene	116	115 (108)
91-20-3	Naphthalene	128	102 (7)
4565-32-6	Benzo(B)Thiophene	134	89 (8)
91-22-5	Quinoline*	129	102 (20)
120-72-9	1H-Indole	117	90 (31)
91-57-6	2-Methylnaphthalene	141	115 (31)
90-12-0	1-Methylnaphthalene	141	115 (28)
92-52-4	Biphenyl	154	153 (35)
208-96-8	Acenaphthylene	152	151 (17)
83-32-9	Acenaphthene	154	153 (93)
132-64-9	Dibenzofuran	168	139 (40)
86-73-7	Fluorene	166	165 (90)
132-65-0	Dibenzothiophene	184	139 (19)
85-01-8	Phenanthrene	178	176 (19)
120-12-7	Anthracene	178	176 (19)
260-94-6	Acridine	179	178 (26)
86-74-8	Carbazole	167	166 (28)
206-44-0	Fluoranthene	202	200 (17)
129-00-0	Pyrene	202	200 (18)
56-55-3	Benzo(A)Anthracene*	228	226 (22)
218-01-9	Chrysene*	228	226 (26)
205-99-2	Benzo(B)Fluoranthene*	252	250 (22)
207-08-9	Benzo(K)Fluoranthene	252	250 (22)
192-97-2	Benzo(E)Pyrene	252	250 (35)
50-32-8	Benzo(A)Pyrene*	252	250 (26)
198-55-0	Perylene	252	250 (24)
193-39-5	Indeno (1,2,3-CD)Pyrene*	276	274 (25)
53-70-3	Dibenz(A,H)Anthracene*	278	279 (20)
191-24-2	Benzo(G,H,I)Perylene*	276	274 (25)
205-82-3	Benzo(J)Fluoranthene*	252	250 (22)

NOTE: The % abundance for the confirmation ion is a typical value. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20% between the calibration standard for any given day and the samples run on that day.

* Carcinogenic PAH as defined in Appendix A of the RAP.

Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. In the FC43 spectra, the ion abundance of masses 131 and 219 are adjusted to a ratio of 1:1. These two ions are then maximized to be approximately 50 to 70 percent of the ion abundance of the base mass 69. This procedure maximizes the sensitivity of the instrument in the mass region of interest for the PAH analysis.

The requirements above will be employed for all compounds in Table 8-1 with the exception of benzo(j)fluoranthene. Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either Benzo(b)fluoranthene or Benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistantly separated by this method. Therefore if present, it will be detected and reported as Benzo(b) and/or Benzo(k)fluoranthene.

8.2 Non-Criteria Analysis

All Non-Criteria analyses will follow the calibration requirements described in CLP Document OLM01.0, or most recent version. In summary, the SOW requires an initial verification that the mass spectrometer is tuned properly using decafluorotriphenyl phosphine (DFTPP). The SOW also requires an initial five-point calibration be performed for all compounds and that this calibration be verified by the analysis of a daily calibration standard.

The calibration requirements in the SOW are based on the determination of a diverse list of semivolatile organics. Calibration is verified on a daily basis by comparing the responses of a few select compounds, termed calibration check compounds (CCC). Only one of these compounds (acenaphthene) is a target PAH for this project. The response of another group of compounds, termed system performance check compounds (SPCC) are used to verify the analytical system is working properly. None of the SPCCs are target PAH for this project. Finally, the target PAH for this project contain compounds not measured under CLP protocols.

Accordingly, the procedures in the SOW for calibration have been modified to accommodate the differences in the monitoring lists. A calibration standard containing all of the analytes shown in Table 8-1 is used for both initial and continuing calibration in place of the CLP standard. The daily calibration is verified by comparing the response of all 32 compounds to the response from the initial calibrations. For the initial calibration, the relative standard deviation (RSD) for <u>each</u> compound must be less than 30 percent or the system is out of control and corrective action must be performed. For continuing calibration, the percent difference for each compound must be less than 30 percent.

The control limit for the daily calibration is based on the accuracy and precision objectives of this project and experience with this group of analytes. The limits in the CLP SOW, which is slightly more stringent, is based on a select group of compounds with extensive method performance data.

8.3 Extended Analysis

In addition to the compounds listed in Table 8-1, the compounds shown in Table 8-2 are required to be determined in the extended monitoring program. This extended list of compounds include phenols and other PAHs specified for this project.

Analyses for the extended list of compounds will be performed on the semivolatiles extract prepared as described in CLP SOW Document OLM01.0, or most recent version.

Since most of the compounds on the extended monitoring list are also target compounds in the CLP protocol, the CLP calibration protocol will be followed.

The system is tuned with DFTPP and calibrated with the semivolatile compounds as specified in the CLP SOW. The compounds used to assess system performance and to verify the continuing calibration (SPCCs and CCCs) are used to verify that the system is in control. The control limits in the SOW are used. The presence of the PAH compounds listed in Table 8-2 is determined by evaluating the library search results generated for the CLP analysis of the sample.

Example retention times, quantitation ions and the internal standards determined at the laboratory for 7,12-dimethylbenz(a)anthracene and 3-methylcholanthrene are listed in Table 8-3.

8.4 Phenolics

A three-point calibration curve covering the linear range of the method will be analyzed prior to the analysis of any samples and with a minimal frequency of once per 12 hours.

TABLE 8-2
TARGET COMPOUNDS FOR EXTENDED ANALYSES

CAS NO.	A. OTHER CARCINOGENIC PAH	REPORTING LIMIT ng/L
195-19-7	Benzo(c)phenanthrene/1	--
215-58-7	Dibenz(a,c)anthracene/2	1.6
192-65-4	Dibenzo(a,e)pyrene/1	--
189-64-0	Dibenzo(a,h)pyrene/1	--
189-55-9	Dibenzo(a,i)pyrene/1	--
57-97-6	7,12-Dimethylbenz(a)anthracene	2.8
56-49-5	3-Methylcholanthrene	3.5

/1 No analytical standards are available.

/2 Coelutes with dibenz(a,h)anthracene. If these isomers are detected, they will be reported as a total value.

CAS NO.	B. ACIDIC COMPOUNDS LISTED IN EPA METHOD 625	REPORTING LIMIT ug/L
108-95-2	Phenol	10
95-57-8	2-Chlorophenol	10
88-75-5	2-Nitrophenol	10
105-67-9	2,4-Dimethylphenol	10
120-83-2	2,4-Dichlorophenol	10
59-50-7	4-Chloro-3-methylphenol	10
88-06-2	2,4,6-Trichlorophenol	10
51-28-5	2,4-Dinitrophenol	50
100-02-7	4-Nitrophenol	50
534-52-1	4,6-Dinitro-2-methylphenol	50
87-86-5	Pentachlorophenol	50

TABLE 8-3

RETENTION TIMES, QUANTITATION IONS AND INTERNAL STANDARDS FOR EXTENDED PAH LIST

Compound	Absolute Retention Time	Relative Retention Time	Quantitation Ions	Internal Standard
7,12-dimethylbenz(a) anthracene	30:51:00 minutes	0.890 minutes	M/Z 256	D_{12}-B(A)P[1] M/Z 264
3-methylcholanthrene	32:48:50 minutes	1.085 minutes	M/Z 268	D_{12}-B(A)P[1] M/Z 264

[1] Benzo(A)Pyrene

9. ANALYTICAL PROCEDURES

9.1 Low-Level Analysis

As specified in the Consent Decree, four types of analyses are to be performed as part of the RAP for this project. These four analyses are defined below, and the details of the specific analytical procedures are presented in subsequent subsections.

- Low-Level: Refers to the determination of a specific list of 21 polynuclear aromatic hydrocarbons using GC/MS with operation in the selected ion monitoring (SIM) mode. The list of target PAH contains carcinogenic and non-carcinogenic compounds and is shown in Table 8-1 of the QAPP. The list includes 14 compounds which are not on EPA's priority pollutant, Appendix IX or Superfund target compound list. The analytical methodology is based on well known principles of GC/MS technology. Although there is no EPA method that embodies this technique for this class of compounds, methods developed for the measurement of polychlorinated dibenzodioxins (e.g., Methods 613 and 8280) are based on selected ion monitoring technology.

- Non-Criteria: The Low-Level PAH method is designed to measure PAH at the sub-ppb level. At higher concentrations, the compounds can be measured under scanning GC/MS conditions. Since scanning GC/MS provides more reliable qualitative data, this method, termed "Non-Criteria PAH" is preferred for samples containing ppb concentrations of PAH. The method is based on the Contract Laboratory Program (CLP) protocol for semivolatile organics with the appropriate modifications to address the differences in the monitoring lists.

- Extended: Some samples are analyzed for the specific list of compounds shown in Table 8-2 of the QAPP using scanning GC/MS. This list, termed "Extended" analyses, includes additional PAH, specific acid (phenolic) compounds and a provision for "identifying" unknown compounds. Unknown compounds will be identified and reported from the analysis of the acid fraction only. As in the Non-Criteria analyses, analyses are performed using CLP protocols with the appropriate modifications.

- Phenolics: Refers to the determination of "total phenols" using a colorimetric procedure.

A method has been developed for the analysis of selected target PAH and heterocycle compounds at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography/mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The method is generally applicable for the measurement of any PAH or related compound. For this project, only those compounds listed in Table 8-1 will be determined.

In summary, a measured volume of sample is extracted with methylene chloride. Analysis of the concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions. Specific details of this methodology can be found in Appendix B, Determination of Low-Level (Part Per Trillion) PAH and Heterocycles in Water. This method is designed to analyze samples containing up to 600 ppt of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the ppb range. However, sample dilutions may result in loss of information concerning recovery of surrogates. For this reason, an optional sample preparation technique is contained in the method. This optional technique can be used if historical information indicates that the target compounds are present in concentrations in excess of 600 ppt.

9.2 Non-Criteria Analysis

The selected target PAH and heterocycle compounds listed in Table 8-1 can be determined by GC/MS in the scanning mode at the ppb and higher concentrations. This analysis, termed Non-Criteria analysis, uses the methodology contained in CLP SOW Document OLM01.0, or most recent version. The major deviations to the semivolatile organic analysis from the SOW are as follows:

1. The calibration is performed as set forth in Section 8 of this QAPP.
2. The internal QC checks are set forth in Section 11 of this QAPP.
3. Data are reported only for those compounds listed in Table 8-1.

9.3 Extended Analysis

The target compounds listed in Table 8-2 are measured using the methodology contained in CLP SOW Document OLM01.0, or most recent version for semivolatile organics. The only deviations from this SOW are as follows:

1. The calibration is performed as described in Section 8 of the QAPP.
2. The only target compound in the analytical reports are those listed in Table 8-2.

9.4 Phenolics

Total phenolics will be determined by RMAL SOP No. 1112 which references Methods 420.1 and 420.2 as published in the "Methods for Chemical Analysis for Water and Waste, EPA 600/4-79-020" (refer to Appendix B).

10. DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction and Validation

All project data will be subjected to a three-tier process including review by operations, by the data review groups for inorganics and GC/MS and the final review by the Project Coordinator prior to its release. The review process has been developed to minimize errors associated with sample processing, sample analysis and data reporting and to ensure that information pertaining to a given sample is well documented.

Appendix A contains Standard Operating Procedures (SOP's) for laboratory data review. Refer to SOP No. LP-RMA-0002 for information relative to review policies and processes. In addition, the SOP's for the analytical methods contain the calculation techniques required to obtain reportable concentrations from the raw data.

10.2 Turnaround Time

In accordance with Section 3.2 of the RAP, RMAL has agreed to a 30 working day turnaround. The City, however, makes no enforceable commitment under the RAP except for a maximum of 5 days from validated time of sample receipt for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, the City makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

10.3 Reporting/Data Deliverables

RMAL shall prepare summary reports and data packages in a format that mimics the format described in CLP SOW Document OLM01.0, or most recent version. Specifically, Form 1, SV-1 and SV-2 in Exhibit B of the CLP SOW will be changed to include the PAH list of parameters shown in Table 8-1 of the QAPP. Form II, SV-1 will show the surrogates for the PAH analysis. Form III, SV-1 will show the spike compounds for the PAH analyses. Form VI, SV-1 and SV-2 and Form VII, SV-1 and SV-2 will be altered to show just the target parameters shown in Table 8-1 of the QAPP. Finally, Form VIII, SV-1 and SV-2 will be modified to show the internal standards for the PAH method. In addition, in the Low-Level PAH analyses, compounds which are determined to be present in the samples based on careful inspection of the data, but which do not meet the secondary ion confirmation criteria will be flagged with an "R". The reporting forms in Exhibit B will be modified to show the target lists of parameters, surrogates and spiking compounds for the Low-Level PAH.

The data packages for total phenolics shall as closely follow CLP deliverables for inorganic analysis as possible. Reports shall contain all applicable CLP forms as well as the associated raw analytical data. The package includes Forms I - III, V and VI (results, initial and continuing calibration verification, blanks, matrix spike and duplicate). The report shall be organized as described in CLP Inorganic SOW 7/88.

RMAL has determined the method detection limits for the part per trillion PAH analysis of water samples, utilizing GC/MS selected ion monitoring, according to the method described in Appendix B to Part 136 of the Friday, October 26, 1984 Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection - Revision 11.1. Table 10-1 lists the compounds, the observed concentrations of seven replicates spiked at 5 parts per trillion, the standard deviations and the method detection limits. RMAL has also determined the method detection limits for part per billion Phenolics according to Method 420.2 as published in the "Methods for Chemical Analysis for Water and Waste, EPA 600/4-79-020" (see Table 10-2).

These calculated method detection limits will be used in sample reporting as follows:

- Analytes detected at concentrations greater than or equal to the calculated method detection limits will be reported with no qualifiers.
- Analytes which are not detected will be reported as the calculated detection limit followed by a "U" qualifier which is used in the EPA Contract Lab Program (CLP) to indicate a non-detected compound.
- Analytes that are detected at concentrations less than the calculated method detection limits will be reported followed by a "J" qualifier which is used in the EPA Contract Lab Program (CLP) to indicate that a reported value is below the method detection limit.

The various items in the data package are listed below:

- Sample Traffic Reports or Chain-of-Custody
- Sample Data Summary Report Including:
  - Case narrative
  - Tabulated target compound results by fraction
  - Surrogate spike analysis results by fraction
  - Matrix spike/matrix spike duplicate results by fraction
  - Blank data by fraction
- Sample Data Package including:
  - Case narrative
  - Traffic reports
  - Raw data

TABLE 10-1
METHOD DETECTION LIMIT REPORT

Compound	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Sample #7	Standard Deviation	Method Detection Limit (3a) *
2,3-Benzofuran	19.4*	20.9*	18.0*	19.5*	20.3*	21.5*	16.6*	1.70*	5.1*
2,3-Dihydroindene	4.3	4.2	4.7	3.7	3.8	4.9	4.7	0.46	1.4
1H-Indene	4.4	4.2	4.6	3.9	4.1	4.7	4.6	0.30	0.9
Naphthalene	20.5*	21.0*	18.5*	20.3*	23.0*	23.5*	17.6*	2.15*	6.5*
Benzo(B)thiophene	3.6	3.5	3.9	3.4	3.3	3.8	4.1	0.29	0.9
Quinoline	4.7	4.0	4.1	3.7	3.3	4.4	4.1	0.45	1.4
1H-Indole	3.7	4.5	5.6	3.2	3.2	4.2	4.0	0.84	2.5
2-Methylnaphthalene	5.4	5.0	5.3	5.1	4.8	4.9	5.7	0.31	0.9
1-Methylnaphthalene	4.5	4.2	4.6	3.8	3.7	4.7	5.2	0.53	1.6
Biphenyl	17.9*	18.1*	16.4*	18.4*	18.1*	19.3*	15.0*	1.43*	4.3*
Acenaphthylene	3.9	3.6	4.6	3.7	3.5	4.4	4.5	0.46	1.4
Acenaphthene	4.2	3.7	4.7	3.5	3.5	4.1	4.1	0.43	1.3
Dibenzofuran	4.3	3.9	4.6	4.1	3.7	4.6	4.2	0.34	1.0
Fluorene	4.4	4.0	4.5	4.0	4.0	4.6	4.8	0.33	1.0
Dibenzothiophene	4.0	3.5	4.0	3.5	3.2	3.9	4.2	0.36	1.1
Phenanthrene	4.7	3.9	4.7	3.9	3.6	4.2	4.5	0.43	1.3
Anthracene	4.5	3.8	4.5	4.1	3.6	4.1	4.6	0.38	1.1
Acridine	4.1	4.3	4.9	4.1	3.8	2.4	2.3	0.98	2.9
Carbazole	4.5	3.2	4.8	3.5	3.9	3.1	3.8	0.64	1.9
Fluoranthene	4.5	3.8	4.7	3.9	3.6	4.4	4.7	0.45	1.4
Pyrene	4.3	3.7	4.4	3.9	3.4	4.2	4.7	0.45	1.4
Benzo(A)anthracene	4.6	3.6	4.0	3.6	3.3	5.3	5.3	0.83	2.5
Chrysene	4.3	3.3	3.7	3.3	2.9	5.1	5.3	0.94	2.8
Benzo(B)fluoranthrene	4.6	3.4	3.8	3.6	2.8	4.9	5.0	0.83	2.5
Benzo(K)fluoranthrene	4.1	3.2	3.5	3.2	3.2	4.9	4.8	0.76	2.3
Benzo(E)pyrene	4.9	3.8	4.1	3.3	3.5	4.9	4.4	0.64	1.9
Benzo(A)pyrene	4.5	3.2	3.8	3.2	2.9	4.8	4.5	0.76	2.3
Perylene	4.6	3.6	3.8	3.5	3.3	5.3	5.1	0.82	2.5
Indeno(1,2,3-CD)pyrene	4.5	3.4	3.4	2.9	3.0	4.5	4.2	0.69	2.1
Dibenz(A,H)anthracene **	4.2	3.5	3.6	3.1	3.3	4.6	4.1	0.54	1.6
Benzo(G,H,I)perylene	3.8	3.0	2.9	2.6	2.9	4.9	4.7	0.94	2.8

Note: Amount spiked = 5 ng/L.

* Data for 2,3-Benzofuran, Naphthalene and Biphenyl were obtained from previous detection limit study. Spike levels = 20 ng/L.

** Compounds co-elute

TABLE 10-2

METHOD DETECTION LIMIT STUDY - TOTAL PHENOLICS

Sample #	Concentration Detected (mg/L)
1	0.0315
2	0.0340
3	0.0291
4	0.0315
5	0.0291
6	0.0291
7	0.0315

Calculated Standard Deviation = 0.0018

Calculated Method Detection Limit = 0.00579 mg/L
= 5.8 ug/L

The City will present reports in a manner consistent with the requirements of the RAP. In addition, data packages containing all elements listed above will be presented for the sample analyses completed, if so directed by the EPA. The EPA shall be responsible for identifying the specific sample analyses for which data packages will be provided.

10.4 Reporting Requirements for Samples Exceeding Advisory Levels or Drinking Water Criterion

For active drinking water wells, RMAL will notify the City of St. Louis Park by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

Parameter	Advisory Level	Drinking Water Criterion
Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene*	3.0 ng/L*	5.6 ng/L
Total Carcinogenic PAH +	15 ng/L**	28 ng/L**
Total Other PAH	175 ng/L	280 ng/L

*Or the detection limit, whichever is largest.

**Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree.

+See Table 10-3.

10.5 Final Evidence Files

The final evidence (or data) files will be maintained for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports, raw data, continuing calibration checks, DFTPP tune, detection limits, chain of custody documentation, quality control data for blanks and matrix spikes, results forms, and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues is included in the package. The City will maintain these files in a secure, limited access area under the custody of the Project Manager. RMAL maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

TABLE 10-3

CARCINOGENIC PAH[a]

benz(a)anthracene
benzo(b)fluoranthene
benzo(j)fluoranthene
benzo(ghi)perylene
benzo(a)pyrene[b]
chrysene
dibenz(a,h)anthracene[b]
indeno(1,2,3-c,d)pyrene
quinoline

(a) The total maximum levels of carcinogenic PAH established in the Consent Decree-RAP are:

Advisory Level - 15 ng/l

Drinking Water Criterion - 28 ng/l

(b) The total maximum levels of the sum of benzo(a)pyrene and debenz(a,h) anthracene are:

Advisory Level - 3.0 ng/l (or the lowest concentration that can be quantified, whichever is greater)

Drinking Water Criterion - 5.6 ng/l

11. INTERNAL QUALITY CONTROL

The internal quality control checks will include field blanks, method blanks, surrogate spikes, duplicate analyses, monitoring of internal standard area, and matrix spike analyses. Each quality control check has a specific level of performance which will be reevaluated in an ongoing basis and amended as appropriate through mutual agreement of the EPA, MPCA, and City. The specific details are presented below.

11.1 Low-Level and Non-Criteria PAH Analyses

Internal quality control checks for the Low-Level and Non-Criteria PAH analyses will consist of method blanks analysis, surrogate compound analysis, matrix spike analysis, analysis of duplicate samples, and monitoring of internal standard areas.

11.1.1 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

Method blank analyses are performed at the rate of one per case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

Different control limits have been established relative to method blanks for the Low-Level and Non-Criteria analyses since the target compounds in Table 8-1 are present as "laboratory contaminants" in method blanks at the ppt concentration level.

For the Low-Level analyses, an acceptable method blank analysis must not contain any carcinogenic PAH in Table 8-1 at concentrations greater than or equal to the Method Detection Limits (MDL) in Figure 10-1 or any non-carcinogenic PAH at a concentration greater than 5 times the MDL. For the Non-Criteria analyses, an acceptable method blank does not contain any PAH in Table 8-1 above 10 micrograms per liter. If the method blanks do not meet these criteria, the analytical system is out of control and the source of the contamination must be investigated and corrective measures taken and documented before further sample analysis proceeds.

* A case is a group or a set of samples collected from a particular site over a given period of time.

11.1.2 Surrogate Compound Analysis

As detailed in the RMAL SOP (Appendix B), the laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compound will be spiked into the sample prior to extraction to measure individual sample matrix effects associated with sample preparation and analysis.

RMAL will take corrective action whenever the surrogate recovery is outside the acceptance criteria shown below. The corrective action is described in Section 15 of this QAPP.

	Acceptance Criteria %	
Surrogate	Low-Level	Non-Criteria
Naphthalene-d8	21-108	37-107
Fluorene-d10	41-162	36-127
Chrysene-d12	10-118	25-160

11.1.3 Matrix Spike/Matrix Spike Duplicate Analysis

Low-Level PAH matrix spike and matrix spike duplicate samples will be analyzed as outlined in the RMAL SOP (Appendix B). Non-Criteria PAH matrix spike and matrix spike duplicate samples will be analyzed pursuant to applicable criteria of CLP SOW Document OLM01.0, or most recent version.

The laboratory will spike and analyze 5% matrix spike and matrix spike duplicate samples. RMAL will spike seven representative compounds into water. These compounds and the spiking levels are listed below:

	Low-Level	Non-Criteria
Naphthalene	10 ng/L	50 ug/L
Fluorene	10	50
Chrysene	10	50
Indene	10	50
Quinoline	10	50
Benzo(e)pyrene	10	50
2-methylnaphthalene	10	50

The matrix spike criteria for data validity are as follows:

- The Matrix Spike - Matrix Spike Duplicate average for each spike compound must fall between the established acceptable limits.

MATRIX SPIKE LIMITS

Compound	Low-Level	Non-Criteria
Naphthalene	20-150	43-128
Fluorene	69-118	51-120
Chrysene	20-132	43-124
IH-Indene	20-150	49-108
Quinoline	20-150	40-126
Benzo(e)pyrene	20-150	20-150
2-methylnaphthalene	20-150	47-138

- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:
  1) 10% for chrysene
  2) 20% for all other compounds.

Corrective action will be performed if these criteria are not achieved as described in Section 15.

11.1.4 Duplicates

Relative percent difference between duplicates will be calculated for each detected compound per procedures outlined in Section 14.3. of this QAPP.

11.1.5 Internal Standard Areas

The area of the internal standard will be monitored on each analysis. The area from the daily calibration standard will be used to set a daily acceptance criteria. If the internal standard areas in samples changes by more than a factor of two (-50 percent to + 100 percent) from the daily standard, corrective action must be performed. Additionally, the retention times of internal standards must agree to +/- 30 seconds of the daily standards.

11.2 Extended Analysis

The internal quality control checks for Extended Analyses will consist of surrogate spikes, matrix spikes, matrix spike duplicates, method blanks, etc. as described in CLP SOW Document OLM01.0, or most recent version. The acceptance criteria are as defined in the SOW.

11.3 Phenolics

The internal quality control checks for phenolics will mimic those for inorganics in the CLP program and will include the analysis of a method blank, a laboratory check standard, a matrix spike sample, a matrix spike duplicate, and a duplicate sample. The specific details for each of these QC checks are summarized below.

11.3.1 Blanks

A "Preparation Blank" is analyzed with each batch of 20 samples. This blank is carried through the entire procedure, including the distillation step. Additional blanks, termed "Initial Calibration Blank" (ICB) and "Continuing Calibration Blank", (CCB) are also analyzed. These blanks are used only to evaluate the determinative step and are not distilled. They are analyzed at a frequency of one ICB per 20 samples and one CCB per 10 samples.

An acceptable blank must not contain phenolics above the nominal reporting limit of 5 micrograms per liter. If any of the blanks contain phenolics above 5 micrograms per liter, the system is out of control and corrective action must be performed.

11.3.2 Laboratory Check Standard

The calibration is verified by the analysis of two different laboratory check standards. An "Initial Calibration Verification" (ICV) check standard is analyzed at a frequency of one per 20 samples. This check is carried through the entire procedure, including the distillation step. The measured value from this check standard must be between 75 percent and 125 percent of the true value.

A "Continuing Calibration Verification" (CCV) check standard is analyzed at a frequency of one per 10 samples. This standard is used to verify the determinative step only. The measured value must be between 85 percent and 115 percent of the true value.

If the measured values from the check standards are not within control limits, the system is out of control and corrective action must be performed.

11.3.3. Matrix Spikes/Matrix Spike Duplicates

As for the other tests, matrix spikes and matrix spike duplicates will be performed at a frequency of 5 percent. The spike level is 50 micrograms per liter. The recovery of the matrix spike must be between 75 percent and 125 percent. Corrective action is performed if these criteria are not achieved.

11.3.4 Duplicates

Field duplicate analyses are performed at a frequency of 10 percent. Corrective action is performed if the relative difference from the duplicate analysis is greater than 70 percent.

12. PERFORMANCE AND SYSTEM AUDITS

The ability of the Sampling Team to successfully monitor pumping wells and monitor wells, and the ability of the laboratory to successfully analyze groundwater samples will be confirmed by a series of audits conducted in conjunction with the implementation of the groundwater monitoring program established in the Consent Decree-RAP.

12.1 Field Audits

EPA Region 5 Central Regional Laboratory (CRL) and the Central District Office (CDO) are responsible for the external audits of field activities, including field sampling and measurements, for compliance of requirements specified for this project. The Quality Assurance Manager and/or Field Team Leader of ENSR will be responsible for internal audits to see if field sampling and measurements are properly followed.

12.2 Laboratory Audits

RMAL participates in a variety of federal and state certification programs, (including the EPA CLP), that subject the laboratory to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data.

12.2.1 External Audits

RMAL will be subjected to EPA performance and system audits for approval/disapproval specific to the requirements of this program. The ~~Laboratory Scientific Support Section (LSSS) of~~ US EPA Region 5 Central Regional Laboratory (CRL) is responsible for the audits.

12.2.2 Internal Audits

In addition to external audits conducted by EPA Region 5 CRL, the City of St. Louis Park and/or Northwest Regional Quality Assurance Manager of ENSR (officed in Fort Collins, Colorado) will be responsible for at least biennial auditing of the RMAL laboratory. Audit procedures will include both system audits and performance audits as necessary to satisfy the City that RMAL is capable of rendering satisfactory laboratory services under this QAPP (see Figure 12-1 for the City of St. Louis Park Audit Checklist).

CITY OF ST. LOUIS PARK AUDIT CHECKLIST

	YES	NO
Sample Receiving		
Are refrigerator/cold storage area temperatures recorded daily and are records properly maintained? Comments:		
Are sample chain-of-custody forms completed properly? Comments:		
Are the temperatures of the coolers being checked and recorded? Comments:		
Are volatile samples stored separately? Comments:		
Is access to sample storage area restricted? Comments:		
Data Review		
Are all calculations checked by the analyst for accuracy and completeness? Comments:		
Are anomalies documented and reported? Comments:		
What corrective actions are taken when the analytical results fail to meet QC criteria? Comments:		
Standard Preparation		
Are Class S weights used to check the balances? Comments:		
Are non-EPA and non-NBS neat materials compared to EPA or NBS whenever possible? Comments:		
Have expired standards and reagents been discarded? Comments:		
Inorganics		
Is the conductivity of the Milli-Q water system checked daily and recorded? Comments:		
Is linearity verified (correlation coefficient of at least 0.995) before sample analysis?		

Figure 12-1 City of St. Louis Park Audit Checklist

Figure 12-1 (continued)

	YES	NO
If the CCV does not meet acceptance criteria, is the system recalibrated and are all affected samples reanalyzed?		

Comments:

Organic Extraction

Are all reagents and solvents screened for potential contamination?
Comments:

What is the source of reagent water?
Comments:

Are spiking solutions and standards prepared from separate stocks?
Comments:

Is glassware cleaned appropriately?
Comments:

Are the hood airflows checked and how often are they checked?
Comments:

GC/MS Lab

Are current SOP's available for all personnel in the area?
Comments:

Is preventive maintenance performed on all instruments?
Comments:

Have MDL studies been performed on all methods?
Comments:

Are method blanks analyzed with every batch of samples?
Comments:
Are results of QC samples verified to determine if QC criteria has been met before sample analysis begins?
Comments:

Are QC results which are outside of acceptance limits checked for error?
Comments:

Are corrective actions taken as necessary and documented and samples reprepped/reanalyzed?
Comments:

Are logbooks reviewed periodically, as indicated by the signature/date/comments of the reviewer?
Comments:

13. PREVENTIVE MAINTENANCE

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. In order to provide this data, RMAL subscribes to the following programs:

- o maintenance agreements/service contracts with instrument manufacturers
- o laboratory preventive maintenance program

13.1 Service Contracts

Analytical equipment utilized by RMAL laboratory personnel for this project are covered by maintenance agreements with the instrument manufacturers. These manufacturers provide for both periodic "preventive" service calls as well as the non-routine or emergency calls.

13.2 Instrument Logbooks

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- o Inventory information:
  equipment name, model number, serial number, manufacturer, date of acquisition, original cost
- o Service tasks and intervals:
  cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience
- o Service record:
  date of breakdown, date of return to service, downtime, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- o calibration/performance checks
- o daily operational notes

Analysts are referred to manufacturers' operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instruments.

Laboratory preventive maintenance includes any tasks that can be performed in-house, i.e., systematic cleaning of component parts as recommended in the instrument manual. If problems cannot be corrected by laboratory personnel, the instrument service representative is contacted and a service call requested to correct the problem.

13.3 Field Equipment

All field equipment shall be inspected daily for damaged or missing pieces, which will be replaced as needed.

13.3.1 Thermometer

The field workers will handle the thermometer with care to preserve its measurement integrity. After each use, the thermometer will be rinsed with de-ionized or potable water, wiped dry, and returned to its protective case.

13.3.2 Water Level Measurement Tape

Before each use, the battery will be checked using the equipment's element test function, and replaced if necessary. The tape and probe will be wiped clean and rinsed with de-ionized or potable water after each use.

13.3.3 Hydrolab

The hydrolab instrument shall be maintained in accordance with the manufacturer's requirements. In particular, the battery will be checked daily, and replaced if necessary. The instrument shall be operated and stored at temperatures above freezing, to avoid damaging the instrument. After each use, the instrument will be rinsed with potable or de-ionized water, wiped dry and returned to its storage container. The sonde unit must be covered with its protective, water-filled cap.

14. SPECIFIC PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to correct, prevent or minimize the recurrence of these events. The quality assessment techniques described below consist of the techniques used to assure that statistical control has been achieved.

The accuracy and precision of sample measurements are influenced by both external and internal factors. External factors or errors are those associated with field collection and sample transportation. Internal factors or errors are those associated with laboratory analysis. External factors are defined briefly in Section 14.1. Internal factors are defined in Section 14.2.

14.1 External Components

The results for quality control samples taken in the field represent the best estimates of accuracy and precision for the samples, since these values reflect the entire process from sample collection through sample analysis. The frequency of these control samples is described in Sections 5 and 6. Below is a brief description of the information provided by each of these control samples:

- Field blank - provides an estimate of bias based on contamination; includes effects associated with sample preservation, shipping, preparation, and analysis.

- Field collected samples or duplicates - independent samples collected at the same point in space and time. These give the best measurement of precision for sample collection through analysis.

14.2 Internal Components

The results of quality control samples created in the laboratory represent estimates of analysis and precision for the preparation and analysis steps of sample handling. This section describes the quality control-type information provided by each of these analytical measurements. The frequency of each of these measurements is discussed in Sections 5 and/or 11.

- Surrogates - provide an estimate of bias based on recovery of similar compounds, but not the compounds analyzed, for each sample, preparation and analysis.

- Internal standard - an analyte that has the same characteristics as the surrogate, but is added to the sample extract just prior to analysis. It measures bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analysis process only.

- Matrix spikes/Matrix spike duplicates - the matrix spike is added prior to preparation and analysis. The analyte used is the same as that being analyzed and usually is added to a selected few samples in a batch of analyses. It incorporates matrix effects associated with the laboratory analysis.

- Method blanks - provide an estimate of bias based on contamination.

14.3 Calculation Techniques

The quality assessment procedures described above require calculations of relative percent difference (duplicate analyses) and percent recovery (matrix and surrogate spikes). The techniques for performing these calculations are described below.

- Precision - is the degree to which the measurement is reproducible. Precision is assessed by duplicate measurements by calculating the Relative Percent Difference (RPD) between duplicate measurements. The RPD is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

D_1 = first sample value

D_2 = second sample value (duplicate)

- Accuracy - is a determination of how close the measurement is to the true value.

  The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where:

X = the observed value of measurement

T = "true" value

- Completeness - is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct normal conditions.

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the data base is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

- Comparability - expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppt, ppb, etc.).

15. CORRECTIVE ACTION

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy;
- Blanks contain target analytes above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department.

Generally, out-of-control events or potential out-of-control events are noted on an out-of-control event form (see Figure 15-1). This form is part of the data package and, thus, must be completed prior to data approval. If an out-of-control event does occur during analysis, for instance, a surrogate recovery falls out the expected range, the analyst must describe on this form: the event, the investigative and corrective action taken, and the cause of the event, and notify the Laboratory Quality Control Director. In some cases, investigation of an out-of-control event will reveal no problems. In such cases, only the event and the investigative action is recorded. If an out-of-control event is discovered during data package review, the Laboratory Quality Control Director notifies the supervisor for corrective action.

PARAMETER _____ QC LOT _____

PROBLEM (Be specific):

_____

_____

_____

ANALYST:_____ DATE:_____

- -

CORRECTIVE ACTION TAKEN, RESULTS OF ACTION:

_____

_____

_____

ANALYST:_____ DATE:_____

- -

SUPERVISOR COMMENTS AND FOLLOW-UP ACTIONS/RESULTS:

_____

_____

_____

SUPERVISOR:_____ DATE:_____

- -

QUALITY ASSURANCE APPROVAL AND COMMENTS:

_____

_____

_____

QA/QC:_____ DATE:_____

Figure 15-1 Out of Control Form

15.1 Low-Level and Extended PAH Analyses

15.1.1 Surrogates

As discussed in Section 11.1.2, corrective action will be performed whenever the surrogate recovery is outside the following acceptance criteria:

Surrogate	Acceptance Criteria %	
	Low-Level	Non-Criteria
Naphthalene-d8	21-108	37-107
Fluorene-d10	41-162	36-127
Chrysene-d12	10-118	25-160

The following corrective action will be taken when required as stated above:

a) Check calculations to assure there are no errors;

b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;

c) If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that an interference specific to the surrogate was present that resulted in the high recovery and this interference would not affect the quantitation of other target compounds. (The presence of this type of interference can be confirmed by evaluating the chromatographic peak shapes and ion intensities of the surrogates.)

d) If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as D with the note surrogate diluted out.

e) Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

15.1.2 Matrix Spikes/Matrix Spike Duplicates

The matrix spike criteria for data validity are as follows:

- The Matrix Spike - Matrix Spike Duplicate average for each spiked compound must fall between the established acceptable limits (refer to Section 11.1.3 for limits).

- Only one compound can be below its required minimum percent recovery.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, the data will be considered valid. Both matrix spike and surrogate spike recoveries will be used in assessing quality assurance/quality control for RMAL's analytical work.

15.1.3 Blanks

If non-carcinogenic PAH are detected in any Low-Level QC method blanks above the MDL but less than 5 times the MDL the corrective action will consist of flagging the data and investigating the source of the problem to implement a corrective action for future work. If the concentration of carcinogenic PAH in the method blank exceeds the MDL or the concentration of non-carcinogenic PAH in the method blank exceeds five times the MDL, additional corrective action, including but not limited to, reanalyses of the blank and reanalyses of the samples may be required.

If target compounds are detected in Non-Criteria method blanks above 10 micrograms per liter, the corrective action will consist of flagging the data and investigating the source of the problem to implement a corrective action for future work.

The relative concentration of compounds in both the samples and the blank are assessed as part of this corrective action. The results of these activities are documented in the narrative.

15.2 Other Corrective Actions

These sections discuss corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis, or in performance and system audits.

15.2.1 Samples

In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all Low-Level (ppt) samples. For all samples, field blanks and matrix spikes and duplicates, subsequent extraction and analysis will be conducted on four intact 1-liter bottles. All field blank duplicates will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported. Additional sample matrix will be required for matrix spike analyses.

If less than four liters of a sample remains after shipment and storage for analysis, the Program Manager will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.2.2 Sample Extracts

If a sample extract is broken or lost during analysis, the Program Manager will be notified and will be responsible for determining the need for replacing the lost sample. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section the action taken.

15.2.3 Quality Control Samples

If a method blank, or matrix spike and its duplicate is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, its duplicate will be analyzed. The analysis report for the sample batch associated with the field blank will clearly note the occurrence in the discussion section.

15.2.4 Performance and System Audits

Each system audit is immediately followed by a debriefing, in which the auditor discusses his findings with the laboratory representatives. The debriefing serves a two-fold purpose. First, laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective strategies, and second, the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his report.

The systems audit report (which may or may not contain performance audit findings) is first issued in draft to the Laboratory Quality Control Director. The QC Director distributes the draft to the Laboratory Director and appropriate supervisors to solicit comments and/or rebuttals. These responses are forwarded, in writing, to the auditor. The auditor makes revisions to the draft, on the basis of these responses, at his discretion. Any points of disagreement between the QA department and the laboratory organization are resolved through discussion before the final report is issued. Written responses to the draft report are attached to the final report as an appendix.

Final audit reports are issued to project management and to corporate management. Items requiring corrective action are documented on a Corrective Action Request Form addressed to the Project Manager. One copy is retained by QA upon issuance. The Project Manager receives the original and one copy. When satisfactory progress has been achieved on each requested action, the Project Manager or designee enters descriptions of actions and results on the form, then retains the copy and returns the original to QA to close the loop.

16. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Executing and administering an effective QA program in a large and complex laboratory system demands the skills of a highly qualified staff. The organizational structure of Enseco's Quality Assurance Group (Fig. 16-1) provides a disciplined national management network which oversees and regulates all laboratory QA functions.

Enseco's Quality Assurance Group is headed by the Corporate Vice President of Quality Assurance, who reports directly to the Enseco Executive Committee and to the Chairman of the Board. This position is responsible for oversight of a program which monitors and controls laboratory operations. This involves the intricate process of developing QA manuals, QC protocols, training programs, Standard Operating Procedures (SOP's), uniform statistical data, interlaboratory and intralaboratory performance evaluation studies, and internal auditing programs. The Corporate Vice President is responsible for the administration and implementation of the QA program at all Enseco laboratories.

Laboratory QA activities are specifically designed to fulfill the requirements of both the individual laboratory and Enseco. Directing these activities is the Division Director who works closely with the laboratory Quality Assurance Director, who in turn enforces and monitors the program.

Because a QA program undergoes its most stringent test at the laboratory level, Laboratory QA Officers hold a cornerstone position in the organizational structure. Enseco QA Officers are highly skilled analytical scientists, knowledgeable in all aspects of laboratory operations. Their responsibilities include diagnosing quality defects and resolving problems with the analytical system; conducting performance evaluation studies, in-house audits, and walk-throughs; performing statistical analyses of data; auditing spike sample results; enforcing chain-of-custody procedures; assisting in the development of QA manual, SOPs and QC protocols; conducting QA training programs; and maintaining extensive records and archives of all QA/QC data.

Laboratory QA Officers report to both the Division Director and to Corporate Vice President of Quality Assurance. They also interface with one another in a peer evaluation and auditing system that encourages assistance and feedback, problem analysis, and collaboration on ways to improve laboratory performance.

In conjunction with the Laboratory QA Department, laboratory vice presidents, directors, and managers are responsible for a subset of QA activities, and work closely with supervisors to evaluate daily laboratory functions.

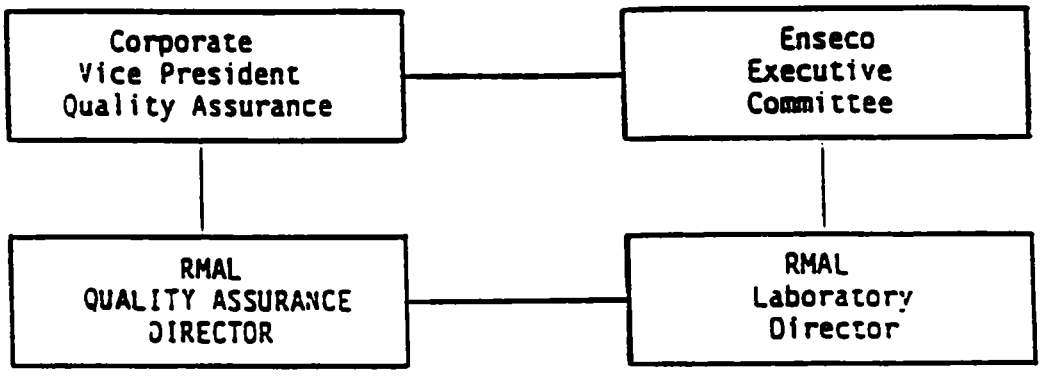


FIGURE 16-1 ENSECO QUALITY ASSURANCE GROUP ORGANIZATION CHART

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Divisional QA Directors submit extensive monthly reports to the VP of QA and the Divisional Director. These reports include:

- The results of all systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations; and
- Comments and recommendations.

In addition, on a weekly basis, a summary of the 5% QA audit of reported data is sent to the Corporate QA Office.

The VP of QA submits weekly reports to the CEO and monthly report to the Enseco Management Committee and each Divisional Director. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

APPENDIX A

STANDARD OPERATING PROCEDURES

INDEX OF STANDARD OPERATING PROCEDURES

SOP NUMBER	SUBJECT	NO. OF PAGES
SOP 7130	Ground-Water Sample Collection from Monitoring Wells	17
SOP 7320	Calibration and Operation of Hydrolab Water Quality Monitor	8
SOP 7510	Packaging and Shipment of Samples	5
LP-RMA-0001	Building Security	2
LP-RMA-0002	Laboratory Data Review	12
LP-RMA-0003	Sample Log-In	8
LP-RMA-0004	Use of Project Assignment Record	29
LP-RMA-0005	Sample Receipt and Chain of Custody	7
	Procedures for Ground Water Monitoring (Minnesota Pollution Control Agency Guidelines)	60

Title: Ground-Water Sample Collection from Monitoring Wells
(REFER TO QAPP SECTION 6.5.4.)

1.0 Applicability

This Standard Operating Procedure (SOP) is concerned with the collection of valid and representative samples from ground-water monitoring wells. The scope of this document is limited to field operations and protocols applicable during ground-water sample collection.

2.0 Responsibilities

The site coordinator or his delegate will have the responsibility to oversee and ensure that all ground-water sampling is performed in accordance with the project-specific sampling program and this SOP. In addition, the site coordinator must ensure that all field workers are fully apprised of this SOP. The field team is responsible for proper sample handling as specified in SOP 7510, Handling and Storage of Samples.

3.0 Supporting Materials

The list below identifies the types of equipment which may be used for a range of ground water-sampling applications. From this list, a project-specific equipment list will be selected based upon project objectives, the depth to ground-water, purge volumes, analytical parameters and well construction. The types of sampling equipment are as follows:

- Purging/Sample Collection

  Bailers
  Centrifugal Pump
  Submersible Pump
  Peristaltic Pump

- Sample Preparation/Field Measurement

  pH Meter
  Specific Conductance Meter
  Filtration Apparatus
  Water-Level Measurement Equipment

Additional equipment to support sample collection and provide baseline worker safety will be required to some extent for each sampling task. The additional materials are separated into two primary groups: general equipment which is reusable for several samplings, and materials which are expendable.

- General

  Project-specific sampling program
  Deionized-water dispenser bottle
  Methanol-dispenser bottle
  Site-specific Health & Safety equipment (gloves, respirators, goggles)
  Field data sheets and/or log book
  Preservation solutions
  Sample containers
  Buckets and intermediate containers
  Coolers
  First-Aid kit

- Expendable Materials

  Bailer Cord
  Respirator Cartridges
  Gloves
  Water Filters
  Chemical-free paper towels
  Plastic sheets

Equipment checklists have been developed to aid in field trip organization and should be used in preparation for each trip.

4.0 Water-Level Measurement

4.1 Introduction

Prior to obtaining a water-level measurement, cut a slit in one side of the plastic sheet and slip it over and around the well, creating a clean surface onto which the sampling equipment can be positioned. This clean working area should be a minimum of eight feet square. Care will be taken not to kick, transfer, drop, or in any way let soil or other materials fall onto this sheet unless it comes from inside the well. Do not place meters, tools, equipment, etc. on the sheet unless they have been cleaned first with a clean rag.

After unlocking and/or opening a monitoring well, the first task will be to obtain a water-level measurement. Water-level measurements will be made using an electronic or mechanical device. Electronic measurement devices will be used in all wells wherein a clearly audible sound cannot be produced with a mechanical device.

4.2 Well Security

Unlock and/or open the monitoring well. Enter a description of condition of the security system and protective casing on the Ground-Water Sample Collection Record shown in Figure 1.

4.3 Measuring Point

Check for the measuring point for the well. The measuring point location should be clearly marked on the outermost casing or identified in previous sample collection records. If no measuring point can be determined, a measuring point should be established. Typically the top (highest point) of the protective or outermost well casing will be used as the measuring point. The measuring point location should be described on the Ground-Water Sample Collection Record and should be the same point used for all subsequent sampling efforts.

4.4. Measurement

To obtain a water-level measurement lower a clean steel, fiberglass tape into the monitoring well. Care must be taken to assure that the water-level measurement device hangs freely in the monitoring well and is not adhering to the wall of the well casing. The water-level measuring tape will be lowered into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to hundredth of a foot) by repeatedly raising and lowering the tape to converge on the exact measurement. The water-level measurement should be entered on the Ground-Water Sample Collection Record. As well point of measurement should be indicated; i.e., top of protective casing, top of pueriser, ground level.

4.5 Decontamination

The measurement device shall be decontaminated immediately after use with a methanol soaked towel. Generally only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface.

5.0 Purge-Volume Computation

All monitoring wells to be purged prior to sample collection. Depending upon the ease of purging, 3 to 10 volumes of ground water to be determined by hydrogeologing prior to sampling present in a well

shall be withdrawn prior to sample collection or one volume if well can be purged dry. The volume of water present in each well shall be computed based on the length of water column and well casing diameter. The water volume shall be computed using Figure 2.

6.0 Well-Purging Methods

6.1 Introduction

Purging must be performed for all ground-water monitoring wells prior to sample collection in order to remove stagnant water from within the well casing and ensure that a representative sample is obtained. The following sections explain the proper procedures for purging and collecting water samples from monitoring wells.

Three general types of equipment are used for well purging: bailers, surface pumps, or down-well submersible pumps.

In all cases pH and/or specific conductance will be monitored during purging. Field parameter values will be entered on the Ground-Water Sample Collection Record along with the corresponding purge volume.

6.2 Bailing

In many cases bailing is the most convenient method for well purging. Bailers are constructed using a variety of materials; generally, PVC stainless steel, and Teflon®. Care must be taken to select a specific type of bailer that suits a study's particular needs. Teflon® bailers are generally most "inert" and are used most frequently. Keep in mind the diameter of each monitoring well so that the correct size bailers are taken to the site. It is preferable to use one bailer per well; however, field decontamination is a relatively simple task if required.

Bailing presents two potential problems with well purging. First, increased suspended solids may be present in samples as a result of the turbulence caused by raising and lowering the bailer through the water column. High solids concentrations may require that total suspended solids (TDS) and the chemical character of solids be evaluated during sample analyses. Second, bailing may not be feasible for wells which require that greater than twenty (20) gallons be removed during purging. Such bailing conditions mandate that long periods be spent during purging and sample collection or that centrifugal pumps be used. All ground-water collected from monitoring wells for subsequent volatile organic compound analyses shall be collected using bailers, regardless of the purge method.

6.3 Surface Pumping

Ground-water withdrawal using pumps located at the ground surface is commonly performed with centrifugal or peristaltic pumps.

All applications of surface pumping will be governed by the depth to the ground-water surface. Peristaltic and centrifugal pumps are limited to conditions where ground water need only be raised through approximately 20 feet of vertical distance. The lift potential of a surface pumping system will depend upon the net positive suction head of the pump and the friction losses associated with the particular suction line, as well as the relative percentage of suspended particulates.

Surface pumping can be used for many applications of well purging and ground-water sample collection. In all cases, pumping cannot be used for the collection of samples to be analyzed for volatile organic compounds (VOCs).

6.3.1 Peristaltic Pump

Peristaltic pumps provide a low rate of flow typically in the range of 0.02-0.2 gallons/min (75-750 ml/min). For this reason, peristaltic pumps are not particularly effective for well purging. Peristaltic pumps are suitable for purging situations where disturbance of the water column must be kept minimal for particularly sensitive analyses. Peristaltic pumps are most often used in conjunction with field filtering of samples and therefore can be used to obtain water samples for direct filtration at the wellhead.

6.3.2 Centrifugal Pump

Centrifugal pumps are designed to provide a high rate of pumping, in the range of 10-40 gallons per minute (gpm), depending on pump capacity. Discharge rates can also be regulated somewhat provided the pump has an adjustable throttle.

When centrifugal pumps are used, samples should be obtained from the suction (influent) line during pumping by an entrapment scheme as shown in Figure 3. Construction of this sampling scheme is relatively simple and will not be explained as part of this SOP. It is suggested that if samples cannot be obtained before going through the pump, that samples be obtained by using a bailer once pumping has ceased. Collecting samples from the pump discharge is not recommended.

6.3.3 Submersible Pump

Submersible pumps provide an effective means for well purging and in some cases sample collection. Submersible pumps are particularly useful for situations where the depth to water table is greater than twenty (20-30) feet and the depth or diameter of the well requires that a large purge volume be removed during purging.

ERT uses the Johnson-Keck pump model SP-81 which has a 1.75 inch diameter pump unit. The pump diameter restricts use to monitoring wells which have inside diameters equal to or greater than two (2) inches. As with other pump-type purge/sample collection methods, submersible pumps will not be used for the collection of samples for analyses of volatile organic compounds. Submersible pumps should never be used for well development as this will seriously damage the pump.

7.0 Sample Collection Procedures

7.1 Bailing

Obtain a clean/decontaminated bailer and a spool of polypropylene rope or equivalent bailer cord. Using the rope at the end of the spool tie a bowline knot or equivalent through the bailer loop. Test the knot for security and the bailer itself to ensure that all parts are intact prior to inserting the bailer into the well.

Remove the protective foil wrapping from the bailer, and lower the bailer to the bottom of the monitoring well and cut the cord at a proper length. Boiler rope should never touch the ground surface at any time during the purge routine.

Raise the bailer by grasping a section of cord using each hand alternately in a "rocking" action. This method requires that the samplers' hands be kept approximately 2-3 feet apart and that the bailer rope is alternately looped onto or off each hand as the bailer is raised and lowered.

Bailed ground water is poured from the bailer into a graduated bucket to measure the purged water volume.

For slowly recharging wells, the bailer is generally lowered to the bottom of the monitoring well and withdrawn slowly through the entire water column. Rapidly recharging wells should be purged by varying the level of bailer insertion to ensure that all stagnant water is removed. The water column should be allowed to recover

to 70-90% of its static volume prior to collecting a sample. Water samples should be obtained from midpoint or lower within the water column.

Samples collected by bailing will be poured directly into sample containers from bailers which are full of fresh ground water. During sample collection, bailers will not be allowed to contact the sample containers.

7.2 Peristaltic Pump

Place a new suction and discharge line to the peristaltic pump. Silicon tubing must be used through the pump head. A second type of tubing may be attached to the silicon tubing to create the suction and discharge lines. Such connection is advantageous for the purpose of reducing tubing costs, but can only be done if airtight connections can be made. Tygon tubing will not be used when performing well purging or collecting samples for organic analysis. The suction line must be long enough to extend to the static ground-water surface and reach further should drawdown occur during pumping.

Measure the length of the suction line and lower it down the monitoring well until the end is in the upper 2-5 inches of the water column present in the well. Start the pump and direct the discharge into a graduated bucket.

Measure the pumping rate in gallons per minute by recording the time required to fill a selected volume of a bucket. Flow measurement shall be performed three times to obtain an average rate.

The pumping shall be monitored to assure continuous discharge. If drawdown causes the discharge to stop, the suction line will be lowered very slowly further down into the well until pumping restarts.

Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record.

Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and Specific Conductance) have stabilized.

When the sample bottles are prepared, each shall be filled directly from the discharge line of the peristaltic pump. Care will be taken to keep the pump discharge line from contacting the

sample bottles. Ground-water samples requiring filtration prior to placement in sample containers, will be placed in intermediate containers for subsequent filtration or filtered directly using the peristatic pump.

At each monitoring point when use of the peristaltic pump is complete, all tubing including the suction line, pump head and discharge line must be disposed of. In some cases where sampling will be performed frequently at the same point, the peristaltic pump tubing may be retained between each use in a clean zip-lock plastic bag.

7.3 Centrifugal Pump

7.3.1 Direct Connection Method (Note: This method requires that the well casing be threaded at the top.)

Establish direct connection to the top of the monitoring well if possible using pipe connections, extensions, and elbows, with Teflon® tape wrapping on all threaded connections. If the centrifugal pump will subsequently be used for sample collection, a sample isolation chamber will be placed in the suction line configuration as shown in Figure 3.

Prime the pump by adding tap water to the pump housing until the housing begins to overflow.

Start the pump and direct the discharge into a graduated bucket or a bucket of known capacity (>2.5 gallons).

Start the pump and measure the pumping rate in gallons per minute by recording the time required to fill the graduated bucket. Flow measurement should be checked periodically to determine if pumping rates are continuous, fluctuating, or diminishing. If discharge stops, the pump will be throttled back to determine if pumping will restart at a lower rate. If pumping does not restart, the pump should be shut off to allow the well to recharge.

Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record. Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and Specific Conductance) have stabilized. Samples should be collected from an in-line discharge valve or with a bailer. The pump should be properly decontaminated between wells.

7.3.2 Down-Well Suction-Line Method

Lower a new suction line into the well. The suction line will have a total length great enough to extend to the water table and account for a minimum of five (5) feet of drawdown. Note should be made that drawdown may exceed the depth where pumping will terminate as a result of a limitation derived from suction-line conditions and the lift potential of the pump. All connections should be made using Teflon® ferrules and Teflon® thread wrapping tape. Run the pump as per Section 7.3.1.

At each monitoring well when use of a centrifugal pump is complete, all suction line tubing should be disposed of properly.

7.4 Submersible Pump

Prior to using a submersible pump, a check will be made of well diameter and alignment. A 1.75 inch diameter decontaminated cylindrical tube should be lowered to the bottom of each monitoring well to determine if the alignment or plumbness of a well is adequate to accommodate the submersible pump. All observations will be entered in the Ground-Water Sample Collection Record.

Slowly lower the submersible pump into the monitoring well taking notice of any roughness or restrictions within the riser.

Count the graduations on the pump discharge line and stop lowering when the stainless steel portion is below the uppermost section of the static water column within monitoring well. Secure the discharge line and power cord to the well casing.

Connect the power cord to the power source (i.e., rechargeable battery pack or auto battery monitor) and turn the pump on (forward mode). When running, the pump can usually be heard by listening near the well head.

Voltage and amperage meter readings on the pump discharge must be checked continuously. The voltage reading will decline slowly during the course of a field day representing the use of power from the battery. Amperage readings will vary depending upon the depth to water table. Amperage readings greater than 10 amps usually indicate a high solids content in the ground water which may cause pump clogging and serious damage. If a steady increase

in amperage is observed, the pump should be shut off, allowed to stop, switched to the reverse mode, stopped again and then placed in forward mode. If high amperage readings persist, the pump should be withdrawn and checked using the large upright cylinder and tap water. Ground-water conditions such as high solids may require that an alternate purge/sample method be used.

Drawdown must also be monitored continuously by remaining near the well at all times and listening to the pump. When drawdown occurs, a metallic rotary sound will be heard as the pump intake becomes exposed and ceases to discharge water, but continues to run. The pump should be lowered immediately to continue pumping water within the uppermost section of the static water column. NOTE: The submersible pump cannot be allowed to run while not pumping for more than five seconds or the pump motor will burn out.

If drawdown continues to the extent that the well is pumped dry, the pump should be shut off and the well allowed to recharge. This on/off cycle may need to be repeated several times in order to purge the well properly.

Measurements of the pumping rate, pH, and specific conductance should be made periodically during well purging. All readings and respective purge volumes should be entered on the Ground-Water Sample Collection Record.

While pumping is on-going and when sample bottles are prepared, bottles will be filled directly from the discharge line of the pump taking care not to touch sample bottles to the discharge line.

At each monitoring well when use of the submersible pump is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures contained in the SOP for Decontamination.

8.0 Sample Preparation

8.1 Introduction

Prior to sample transport or shipment, ground-water samples may require filtration and/or preservation dependent on the specific type of analysis required.

Specific preservation techniques are described in the EPA document, Handbook for Sampling and Sample Preservation of Water and Wastewater (EPA-600/4-82-029). The EPA manual and laboratory manager should be consulted during the planning stage of the project. Project-specific sampling plans shall be assembled using the approved procedures obtained from the EPA manual.

8.2 Filtration

Ground-water samples collected for dissolved metals analyses will be filtered prior to being placed in sample containers. Ground-water filtration will be performed using a peristaltic pump and a 0.45 micron, water filter. Typically the water filters are 142 mm in diameter and are usually placed in 142 mm polycarbonate housings.

The filtration of ground-water samples shall be performed either directly from the monitoring well or from intermediate sample containers such as decontaminated buckets. In either case, well purging shall be performed first. Fresh ground water shall then be filtered and discharged from the filtration apparatus directly into sample containers. For most dissolved metal analyses, pH adjustment of the sample is also required and shall be performed after filling the sample bottles. This is generally accomplished using laboratory supplied compounds such as sulfuric or nitric acid and sodium hydroxide.

9.0 Documentation

A number of different documents must be completed and maintained as a part of ground-water sampling effort. The documents provide a summary of the sample-collection procedures and conditions, shipment method, the analyses requested and the custody history. The list of documents is:

- Ground-water sample collection record
- Sample labels
- Chain of custody forms and tape
- Shipping receipts

Sample labels shall be completed at the time each sample is collected and will include the information listed below. A sample label is shown in Figure 4.

- Client or project name
- Sample number
- Designation (i.e., identification of sample point no.)
- Analysis
- Preservative (e.g., filtration, acidified pH<2 HNO_3)
- Sample-collection date
- Sampler's name

Figure 5 displays the chain of custody record used by ERT. The chain of custody form is the record sample collection and transfer of custody. Information such as the sample collection date and time of collection, sample identification and origination, client or project name shall be entered on each chain of custody record. In accordance with 40 CFR 261.4(d) the following information must accompany all ground water samples which are known to be non-hazardous and to which U.S. Department of Transportation and U.S. Post Office regulations do not apply. Such information is:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of each sample,
- date of shipment, and
- description of sample.

The chain of custody forms provide a location for entry of the above-listed information.

10.0 References

EPA, Handbook for Sampling and Sample Preservation of Water and Wastewater EPA-600/4-82-029, September 1982.

Geotrans, Inc. RCRA Permit Writer's Manual, Ground-Water Protection prepared for U.S. EPA. Contract No. 68-01-6464, October 1983.

Code of Federal Regulations, Chapter 40 (Section 261.4(d).

Figure 1

ENSR

Well No. _____

GROUND WATER SAMPLE COLLECTION RECORD

Job No. _____ Date: _____

Location: _____ Time: S _____

Weather Conds.: _____ F _____

1. WATER LEVEL DATA: (from ToC) ToC Elevation (from LS) _____

a. Total Well Length (+ TC) _____ (known, meas.) Tape Corr. (TC) _____

b. Water Table Elev. (+ TC) _____ Well Dia. _____

c. Length of Water Column _____ (a-b)

2. WELL PURGING DATA:

a. Purge Method _____

b. Required Purge Volume (@ ____ well volumes) _____

c. Field Testing: Equipment Used _____

Volume Removed	T°	PH	Spec. Cond.	Color

3. Sample Collection: Method _____

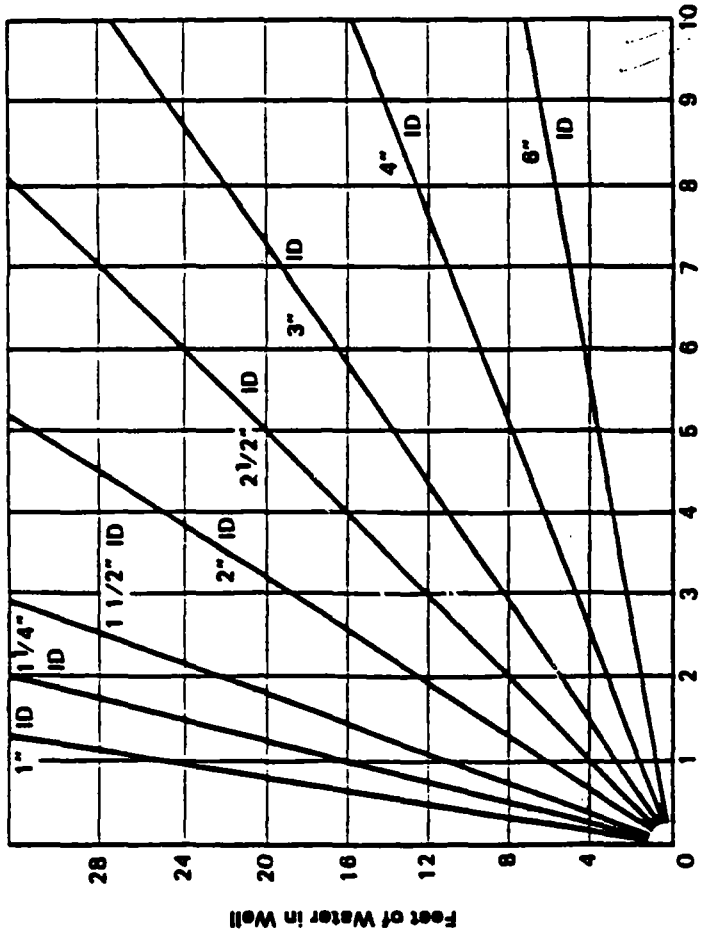
Container Type	Preservation	Analysis Req.

Comments: _____

1985-12-84

Volume/Linear Ft. of Pipe		
ID(in)	Gal	Liter
1/4	0.003	0.010
3/8	0.006	0.022
1/2	0.010	0.039
3/4	0.023	0.087
1	0.041	0.164
2	0.163	0.618
3	0.367	1.39
4	0.653	2.47
6	1.47	5.56

(b) Volume Factors



(a) Graphical Explanation

Figure 2 Purge Volume Computation

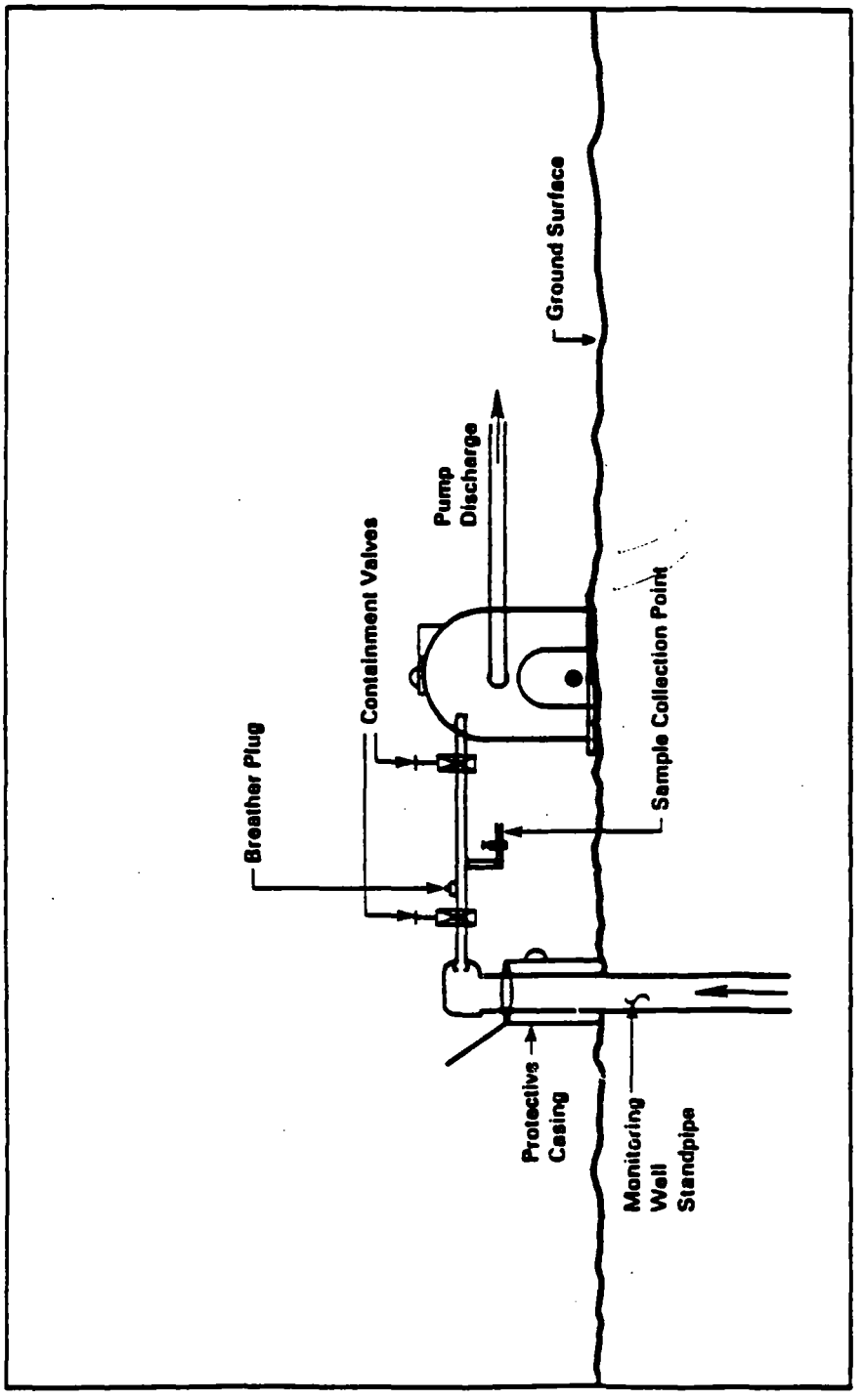


Figure 3 Down Well Suction Line Configuration

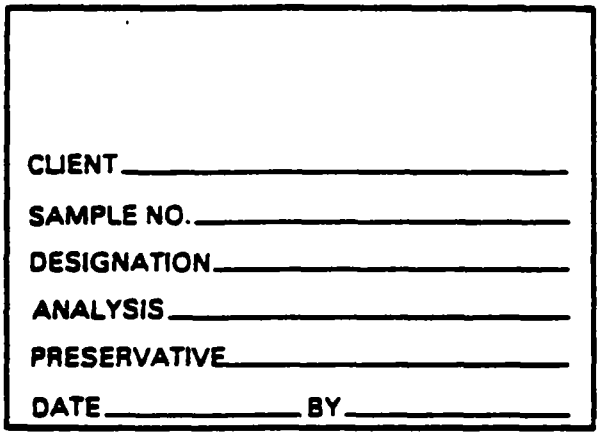


Figure 4 Sample Container Label

CHAIN OF CUSTODY RECORD

Client/Project Name	Project Location	ANALYSES
Project No.	Field Logbook No.	
Sampler: (*Signature*)	Chain of Custody Tape No.	

Sample No./ Identification	Date	Time	Lab Sample Number	Type of Sample							REMARKS

Relinquished by: (*Signature*)	Date	Time	Received by: (*Signature*)	Date	Time
Relinquished by: (*Signature*)	Date	Time	Received by: (*Signature*)	Date	Time
Relinquished by: (*Signature*)	Date	Time	Received for Laboratory: (*Signature*)	Date	Time
Sample Disposal Method:	Disposed of by: (*Signature*)			Date	Time
SAMPLE COLLECTOR	ANALYTICAL LABORATORY				
				No	

1974-3-84

Figure 5 Sample Chain-of-Custody Record

STANDARD OPERATING PROCEDURE
(REFER TO QAPP SECTION 6.7)

Title: Calibration and Operation of Hydrolab Water Quality Monitor

Date: 1st Qtr 19
Number: SOP 7320
Revision: 1

1.0 Applicability

This Standard Operating Procedure (SOP) provides basic instructions to be employed for the field operation of Hydrolab digital multimeters (Model Nos. 4041 and 8000). Hydrolabs are used for field measurement of water-quality parameters.

2.0 Responsibilities

The field team is responsible for ensuring that the Hydrolab unit is in proper operating condition prior to use in the field. All system-calibration checks are the responsibility of the field team.

3.0 Materials

- Hydrolab Operation and Maintenance Instruction Manual
- Hydrolab Sonde unit, battery pack and surface unit
- Hydrolab calibration-cup
- Two Fisher-brand laboratory potassium chloride (KCl) standard solutions (known conductivity at 25°C)
- Two freshly prepared pH buffer solutions. Generally pH 7.0 and pH 4.0 or 10.0 are used.
- Distilled or de-ionized water (approximately two liters)
- Chemical-free paper towels
- Screwdrivers (as supplied in the Hydrolab Accessory Kit)

4.0 Procedures

The Hydrolab provides simultaneous measurement of four water quality parameters; 1) dissolved oxygen, in mg/l, 2) temperature, in °C; 3) pH, in standard units, and 4) conductivity, in umhos/cm (uS/cm). The panel switch on the front of the indicator unit controls which parameter is being measured and read-out.

The display is read in the following manner; temperature, pH and dissolved oxygen are read out directly. For example, a temperature of 21.8°C will be displayed as 21.8. A dissolved oxygen (D.O.) or pH reading of 8.1 will be displayed at 08.1. Conductivity is read out directly on the 2k scale. If the 20k scale is required to measure higher conductivity the number that is displayed will need to be

multiplied by 10. In the 200k scale the reading will be multiplied by 100. For example, suppose the sample being measured has a conductivity of 1527 uS/cm. Using the 2k scale, the display will show 1527 (direct read-out). Using the 20k scale the display will show 153 (153 x 10 = 1530 uS/cm). Using the 200k range the display will show 015 (015 x 100 = 1500 uS/cm). Only the Hydrolab model 4041 offers the three scale measurement. The Hydrolab model 8000 is restricted to measurement of conductivity within the range of 0-2000.

4.1 Hydrolab Calibration

A complete calibration check should be performed before going to and after returning from a field sampling/water quality measurement activity. The calibration procedures should be carried out in a controlled environment such as a laboratory, but a field office or closed-in shelter may also be used.

At least one hour prior to calibration, take the following preparatory steps:

1) Remove the "Storage-Cup" from the Sonde Unit.

2) Remove the protective guard from the dissolved oxygen sensor.

3) Install the "Calibration-Cup" on the Sonde Unit and fill to the brim with distilled water.

4) Seal the Calibration Cup with the soft plastic cap and store the sonde unit, calibration standards, and the distilled water at constant room temperature for at least one hour in order to bring the various sensors, temperature compensating elements, and the calibration solutions into thermal equlibrium (within a few degrees).

All of the calibration controls are located on the front panel of the Indicator Unit. Adjustments, if necessary, should be made in the following manner:

1) Remove the appropriate seal-screw for the parameter being adjusted.

2) Insert a small screwdriver through the access hole and adjust the calibration control in the direction which brings the reading into agreement with the value of the standard solution being employed.

3) Replace the seal-screw.

A RINSE STEP will be used several times during the calibration procedure. It is to be performed in the following manner: Fill the calibration cup halfway with de-ionized or distilled water. Snap on the soft plastic cap; shake the sonde unit for ten seconds and then pour out the water. Repeat twice more using fresh de-ionized or distilled water. Remove the cup and shake as much of the rinse water as possible from the electrodes.

4.1.1 Dissolved Oxygen Calibration

The Dissolved Oxygen system is the first to be calibrated since the water that has been stored in the calibration cup is used to maintain control of the temperature inside the cup. The calibration standard is either a water sample of a known D.O. concentration (determined in the laboratory by the Winkler or iodometric method in accordance with Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA-AWWA-WPCF, 1980 or water-saturated air at the temperature inside the calibration cup. The following procedures are for the water-saturated air method for D.O. calibration.

Invert the Sonde Unit and remove the soft plastic cap. Pour off enough water to bring the level to just below the D.O. membrane- retainer O-ring. With a clean paper towel or tissue blot any moisture from the D.O membrane. Cover the calibration cup mouth with one of the hard plastic caps provided in the Accessory Kit. This will keep drafts from blowing on the membrane. Do not seal the cup with the plastic cap, because that could cause a partial-pressure change in the cup. Wait approximately 5 minutes, or until the reading is stable, then switch to the TEMPERATURE position and record the temperature reading. Refer to Table 1 for the correct oxygen concentration at this temperature. Since the table values refer to concentrations at Standard Pressure it will be necessary to correct the value for local barometric pressure. This should be done in the following manner:

Correct D.O. Setting = (Local Barometric Pressure/760mm) x (Table value at Cup Temperature)

EXAMPLE: If T = 28.5°C and Local Barometric Pressure = 800mm,

Correct D.O. Setting = (800mm/760mm) x (7.6 mg/l)
= 8.0 mg/l

If a barometer is not available, the equivalent pressure may be estimated from Table 2 which relates atmospheric pressure with elevation above mean sea level. Therefore, the approximate atmospheric pressure at an altitude of 2000 feet, for example, would be: Local Atmospheric Pressure = 705mm Hg.

Adjust the Dissolved Oxygen calibration control until the proper value (rounded to nearest tenth) is displayed. Pour our the water; and then follow with a RINSE STEP.

4.1.2 pH Calibration

Calibrating the pH system requires the use of two Fisher-brand pH laboratory buffer solutions. Depending upon the application, either pH 4.0 or pH 10.0 is used in addition to pH 7.0. Invert the sonde unit and fill the calibration cup with fresh pH 7.0 buffer solution. Switch to "pH", and wait approximately 5 minutes for thermal equilibrium. Then adjust the pH calibration control until 7.0 is displayed on the read-out.

Pour out the 7.0 buffer and repeat the RINSE STEP. Invert the sonde unit and screw on the calibration cup; fill with 10.0 or 4.0 buffer. After approximately 5 minutes, adjust the pH "Slope" control until either 10.0 or 4.0 (as appropriate for the buffer being used) is displayed on the read-out. Pour out the buffer and repeat the RINSE STEP <u>Two Times</u>

4.1.3 Conductivity Calibration

After the second RINSE STEP, take a clean paper towel or tissue, and blot most of the moisture in the electrode area so that the standard will not suffer dilution.

Install a clean calibration cup and invert the sonde unit. The conductivity system is calibrated using at least two prepared KCl standard solutions with a known conductivity at 25°C. From Table 3, select two standard solutions with values of approximately one-third and two-thirds of the range you are most likely to encounter in the field. For example, if you are going to be working in fresh water (0-2K scale; you would want to use a 0.01M standard and a 0.005M standard. Select the <u>more concentrated</u> of the two standards and pour it slowly down the side of the calibration cup until full. When the reading is stable, adjust the conductivity calibration control until the display matches

the value listed in Table 3. Empty the calibration cup and repeat the RINSE STEP Two Times. Pour in the second standard. Check the reading on the Display. It should be correct within ± 1% of the range being used. For example, if the 0-2K scale is used, the reading for the second standard should be correct within ± 20 uS/cm of the true value. Pour out the standard solution. Perform a RINSE STEP.

4.1.4 Temperature Calibration

The temperature system is factory calibrated and is accurate to ± 0.2°C. No calibration adjustment is provided. A periodic check of the temperature system against an NBS-traceable thermometer should be performed as a verification.

4.2 Final Preparation

Turn the system off and disconnect the system components. Replace all rubber dust caps. Remove the Calibration Cup from the Sonde Unit and replace the protective guard on the dissolved oxygen electrode. Fill the Storage Cup with tap water and install onto the Sonde Unit. The system is now calibrated and ready for field use.

4.3 Field Operation

Remove the Storage Cup from the calibrated sonde unit and install the guard or the optional sample circulator. Connect the system components. Lower the sonde unit into the water (sideways, if possible) and shake it to dislodge air bubbles trapped in the conductivity cell block. Release the sonde unit and lower it to sample depth. Wait until the readings stabilize (D.O. is the best indicator) and then record the value for each parameter. Repeat at new depths or locations.

When using for ground water sampling, pour/place a sample of ground water into the Storage Cup and attach it to the sonde so that all nodes are submerged.

Check the battery voltage occasionally; charge or change batteries if the level drops below 10.5 volts. DO NOT charge the battery routinely after each day's use. Doing so may shorten the life of the battery. Use the battery until the voltage level drops to between 10.5 and 11.0 volts. At this point put the battery on charge for 24 hours.

TABLE 1

DISSOLVED OXYGEN SATURATION VALUES IN DISTILLED WATER AT 760 mm Hg

Temp. (°C)	DO (mg/l)	Temp (°C)	DO (mg/l)
0.0	14.6	15.5	9.9
0.5	14.4	16.0	9.8
1.0	14.2	16.5	9.7
1.5	14.0	17.0	9.6
2.0	13.9	17.5	9.5
2.5	13.7	18.0	9.4
3.0	13.5	18.5	9.3
3.5	13.3	19.0	9.2
4.0	13.1	19.5	9.1
4.5	13.0	20.0	9.0
5.0	12.8	20.5	8.9
5.5	12.6	21.0	8.8
6.0	12.5	21.5	8.8
6.5	12.3	22.0	8.7
7.0	12.1	22.5	8.6
7.5	12.0	23.0	8.5
8.0	11.8	23.5	8.4
8.5	11.7	24.0	8.3
9.0	11.6	24.5	8.2
9.5	11.4	25.0	8.2
10.0	11.3	25.5	8.1
10.5	11.1	26.0	8.0
11.0	11.0	26.5	8.0
11.5	10.9	27.0	7.9
12.0	10.8	27.5	7.8
12.5	10.6	28.8	7.7
13.0	10.5	28.5	7.6
13.5	10.4	29.0	7.6
14.0	10.3	29.5	7.5
14.5	10.2	30.0	7.4
15.0	10.0	30.5	7.4

TABLE 2

Site Elevation (Feet above mean sea level)	Approximate Mean Barometric Pressure (mm Hg)
1000	733
1500	720
2000	705
2500	694
3000	680
3500	669
4000	656
4500	644
5000	632
5500	620
6000	609
6500	598
7000	586
7500	575
8000	564
8500	554
9000	543
9500	533
10000	523

TABLE 3

CONDUCTIVITY CALIBRATION STANDARDS

Conducitivies of Potassium Chloride Solutions at 25°C M.W. = 74.555			Conductivity Reading on Hydrolab Display for Given Range Setting (uS/cm)		
Conc.	Grams KCl/L	uS/cm	(0-2K)	(0-20K)	(0-200K)
0.0005	0.03728	73.9		-	-
0.001	0.07456	147.0	147	-	-
0.002	0.1491	292.0	292	-	-
0.005	0.3728	717.8	718	-	-
0.01	0.7456	1.413K	1413	141	-
0.02	1.491	2.767K	---	277	-
0.05	3.728	6.668K	---	667	-
0.1	7.456	12.90K	---	1290	129
0.2	14.911	24.82K	---	---	248
0.5	37.278	58.64K	---	---	586
1.0	74.555	111.9K	---	---	1119

NOTES:

(1) Two conductivity standards are recommended for each range setting (boxed-in values). Calibration adjustments will be made first with the higher concentration and then with the lower concentration.

(2) Single dashes indicate ranges which are not recommended for calibration checks.

(3) The Hydrolab model 8000 is restricted to conductivity readings between 0-2000 µS/cm (0-2k) scale), therefore conductivity readings and thus calibration solutions within the 0-20k and 0-200k ranges will not apply.

Title: Packaging and Shipment of Samples
(REFER TO QAPP SECTION 6.6)

1.0 Applicability

This Standard Operating Procedure (SOP) is concerned with procedures associated with the packaging and shipment of samples. Two general categories of samples exist: environmental samples consisting of air, water and soil; and waste samples which include non-hazardous solid wastes and hazardous wastes as defined by 40 CFR Part 261.

2.0 Responsibilities

It is the responsibility of the project manager to assure that the proper packaging and shipping techniques are utilized for each project. The site operations manager shall be responsible for the enactment and completion of the packaging and shipping requirements outlined in the project specific sampling plan. The site operations manager shall be responsible to research, identify and follow all applicable U.S. Department of Transportation (DOT) regulations regarding shipment of materials classified as waste.

3.0 General Method

The objective of sample packaging and shipping protocol is to identify standard procedures which will minimize the potential for sample spillage or leakage and maintain field sampling program compliance with U.S. EPA and U.S. DOT regulations.

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when all of the following conditions are applicable:

A. Samples are being transported to a laboratory for analysis;
B. Samples are being transported to the collector from the laboratory after analysis;
C. Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

Qualification for categories A and B above require that sample collectors comply with U.S. DOT and U.S. Postal Service (USPS) regulations or comply with the following items if U.S. DOT and USPS regulations are found not to apply:

The following information must accompany all samples and will be entered on a sample specific basis on chain of custody records:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of sample,
- date of shipment,
- description of sample, and

in addition, all samples must be packaged so that they do not leak, spill or vaporize.

4.0 General Methods

4.1 Place plastic bubble wrap matting over the base and bottom corners of each cooler or shipping container as needed to manifest each sample.

4.2 Obtain a chain of custody record as shown in Figure 1 and enter all the appropriate information as discussed in Section 3.0 of this SOP. Chain of custody records will include complete information for each sample. One or more chain of custody records shall be completed for each cooler or shipping container as needed to manifest each sample.

4.3 Wrap each sample bottle individually and place standing upright on the base of the appropriate cooler, taking care to leave room for some packing material and ice or equivalent. Rubber bands or tape should be used to secure wrapping, completely around each sample bottle.

4.4 Place additional bubble wrap and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler.

4.5 Place ice or cold packs in heavy duty zip-lock type plastic bags, close the bags, and distribute such packages over the top of the samples.

4.6 Add additional bubble wrap/styrofoam pellets or other packing materials to fill the balance of the cooler or container.

4.7 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain of custody form. Sign and date the chain of custody tape.

4.8 To complete the chain of custody form enter the type of analysis required for each sample, by container, under the "ANALYSES" section. Under the specific analysis enter the quantity/volume of sample collected for each corresponding analysis.

If shipping the samples where travel by air or other public transportation is to be undertaken, sign the chain of custody record thereby relinquishing custody of the samples. Relinquishing custody should only be performed when directly transmitting custody to a receiving party or when transmitting to a shipper for subsequent receipt by the analytical laboratory. Shippers should not be asked to sign chain of custody records.

4.9 Remove the last copy from the chain of custody record and retain with other field notes. Place the original and remaining copies in a zip-lock type plastic bag and place the bag on the top of the contents within the cooler or shipping container.

4.10 Close the top or lid of the cooler or shipping container and with another person rotate/shake the container to verify that the contents are packed so that they do not move. Improve the packaging if needed and reclose.

When transporting samples by automobile to the laboratory, and where periodic changes of ice are required, the cooler should only be temporarily closed so that reopening is simple. In these cases, chain of custody will be maintained by the person transporting the sample and chain of custody tape need not be used. If the cooler is to be left unattended, then chain of custody procedures should be enacted.

4.11 Place the chain of custody tape at two different locations on the cooler or container lid and overlap with transparent packaging tape. For coolers with hinged covers, if the hinges are attached with screws, chain of custody tape should also be used on the hinge side.

4.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of one to two full wraps of packaging tape will be placed at at least two places on the cooler. Shake the cooler again to verify that the sample containers are well packed.

4.13 If shipment is required, transport the cooler to an overnight express package terminal or arrange for pickup. Obtain copies of all shipment records as provided by the shipper.

4.14 If the samples are to travel as luggage, check with regular baggage.

4.15 Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and will sign "received by laboratory" on each chain of custody form. The laboratory will verify that the chain of custody tape has not been broken previously and that the chain of custody tape number corresponds with the number on the chain of custody record. The analytical laboratory will then forward the back copy of the chain of custody record to the sample collector to indicate that sample transmittal is complete.

5.0 Documentation

As discussed in Section 4.0 the documentation for supporting the sample packaging and shipping will consist of chain of custody records and shipper's records. In addition a description of sample packaging procedures will be written in the field log book. All documentation will be retained in the project files following project completion.

0908J

CHAIN OF CUSTODY RECORD

Client/Project Name	Project Location	ANALYSES
Project No.	Field Logbook No	
Sampler: (*Signature*)	Chain of Custody Tape No.	

Sample No./ Identification	Date	Time	Lab Sample Number	Type of Sample							REMARKS

Relinquished by: (*Signature*)	Date	Time	Received by: (*Signature*)	Date	Time
Relinquished by: (*Signature*)	Date	Time	Received by: (*Signature*)	Date	Time
Relinquished by: (*Signature*)	Date	Time	Received for Laboratory: (*Signature*)	Date	Time
Sample Disposal Method:	Disposed of by: (*Signature*)			Date	Time

SAMPLE COLLECTOR

ERT - A Resource Engineering Company
696 Virginia Road
Concord, MA 01742
617-369-8910

ANALYTICAL LABORATORY

ENSR

Nº 1663

1974 3-84

Figure 1

Enseco

STANDARD
OPERATING
PROCEDURE

Subject or Title:
BUILDING SECURITY - REFER TO QAPP SECTION 7

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0001	Original	12/9/87

Supersedes:

1. Purpose:

The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.

2. Policies:

RMAL's security policy includes controlled access to the building, testing areas and data files, confidentially agreements with all personnel, identification badges for all personnel, electronic security and fire alarm systems, and a security guard. All visitors are also assigned visitor badges and are accompanied by RMAL staff during their stay in the facility.

3. Safety Issues: Not Applicable

4. Procedure:

Building Security

a. All exterior doors to the facility will remain locked at all times with the exception of the front entrance.
b. During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period.
c. During the hours of 6:00 p.m. to 7:00 a.m., the front entrance is controlled by security guard. All persons entering or leaving the facility will be recorded by the security guard. The alarm system is activated during this time period to prevent all other exterior doors from being usable, including sample receiving and the patio doors.

Prepared by: Allen J. Hedrick	Date:
Management Approval: Paul Wilds	Date: 12/10/87
QA Officer Approval: Robert C Hamisch	Date: 12/9/87

d. Sample receiving during the hours of 6:00 p.m.to 7:00 a.m. is permitted only with the assistance of the security guard.

Personnel Identification

a. All employees and visitors are required to wear security badges at all times while on the premises of all ENSECO divisions.
b. The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.
c. The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an ENSECO employee before access to any building will be allowed.

Building Alarm System

a. While it is not anticipated that employees will have to set or disarm the alarm system, it is important that employees understand the procedure. Unless used correctly, the alarm will go off and the Arvada Police Department will be called.
b. The procedure is confidential information and can be obtained from the Personnel Department.

5. Responsibilities:

a. It is the responsibility of each employee to maintain confidentiality of all clients data.
b. The Personnel Department is responsible for issuing employee identification badges and having signed "Confidentiality Agreements" in each employee's personnel file.
c. The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours. The security guard is likewise responsible for visitor and employee comings and goings between the hours of 6:00 p.m. and 7:00 a.m.
d. Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

6. Comments:

STANDARD
OPERATING
PROCEDURE

Subject or Title: Laboratory Data Review - REFER TO QAPP SECTION 10.1		Page 1 of 12
SOP No.: LP-RMA-0002	Revision No.: Original	Effective Date: 12/9/87
Supersedes:		

1. Purpose

All laboratory data will be subjected to a rigorous data review process prior to its release to the client. The review process has been developed to minimize errors associated with sample processing, sample analysis and data reporting and to ensure that information pertaining to a given sample is well-documented. The process consists of a three-level review whereby results generated for a specific project are evaluated to ensure that

- o project is complete;
- o precision, accuracy and detection limits are met;
- o raw data interpretation is correct;
- o all calculations are correct;
- o contractual requirements are met; and,
- o all information is well documented for archival purposes.

Enseco/RMAL uses a computerized Laboratory Information Management System (LIMS), as well as a variety of custom software programs designed to perform calculations, check results, generate reports, and to ensure data integrity and security. Whenever possible, historical client-specific data may aid in the review process as an additional check on generated results.

2. Policies

All project data will be subjected to a three-tier review process including review by operations, the data review group for inorganics, GC/MS, and chromatography and the final review by the project or client managers. Data will not be released to the client until the review process is completed.

Prepared by: Allen J. Medine, Ph.D.	Date: December 9, 1987
Management Approval:	Date: 12/10/87
QA Officer Approval: Robert E. Hamisch	Date: 12/10/87

3. Safety Issues

There are no direct safety issues which are of concern for the data review process. As with other non-analytical activities, caution should always be exercised when performing data review functions in the laboratory. For example, discussing problems with analysts, examining original samples, checking preparation aliquots will require review personnel to be in the laboratory or in appropriate storage areas. A review of safety concerns for all of these areas shall be implemented.

4. Procedure

The data review framework is essentially the same for the metals, non-metals, GC/MS and chromatography groups. The differences between each groups procedure are due to analysis differences, data entry and data correction software developed for LIMS. The data review process consists of three levels (LEVEL 1, LEVEL 2 and LEVEL 3). The general framework for the laboratory review process is shown in Figure 1.

A. LEVEL 1 REVIEW

The LEVEL 1 REVIEW begins at the analytical (bench) stage where LEVEL 1 review is primarily a self-review of all information generated during the analysis. During the analytical test, the analysts have much information concerning the precision, accuracy and problems. The intent of the data review program is to take advantage of this condition by review of all analytical details generated by the analyst and subsequent approval of the test results and QC by the analysts immediate supervisor. Specifically, the functions of the analyst and supervisor are as follows:

ANALYST:

1. Review Prep Lab Notes - Preparation lab notes are to be reviewed to determine if there were anomalies observed which may affect the analysis for certain parameters.

2. Review Special Instructions - For certain projects, the Client may have specified certain modifications to a standard test, analysis using a custom test, project specific QC, or special preparation of the sample.

3. Record All Necessary Information - While this may be considered more of an operations or analytical method concern, proper documentation of the analysis, in sufficient detail to allow re-creation of the analysis, is essential for an effective, efficient data review program and to permit development of a sound data archive program. An important part of data recording is to reveal whether

the test proceeded according to the Analytical Method SOP and that deviations from the method, anomalies during analysis, or that decisions concerning re-analysis are well-documented.

4. Check All Calculations - Errors frequently occur during calculations for standard curves, dilution factors, unit conversions, or extrapolations from instrument response to appropriate concentrations. The analysts will check ALL calculations, or verify data entry into software designed to perform calculations, examine results for agreement with expected results (i.e. order of magnitude or better) and indicate that calculations were reviewed on the LEVEL 1 REVIEW CHECKLIST.

5. Provide Data and QC Summary - Summaries of parameter concentrations and QC data generated are to be provided to the supervisor along with raw data (bench sheets, chromatograms, etc.) for supervisor approval of the analytical results.

6. Provide Out of Control/Anomally Sheet - Information regarding out of control situations or anomalies is necessary for review personnel to re-create the analysis when there are questions concerning the data which has been generated during the analysis. Holding time violations are to be clearly indicated along with the appropriate reasons for the violation.

7. LEVEL 1 REVIEW CHECKLIST - The function of the checklist is to indicate that the above items have been considered in the analysis. The LEVEL 1 REVIEW CHECKLIST is shown in Figure 2. There are more detailed items which are considered during the analysis and the review procedure by both analysts and the immediate supervisor in the GC/MS, GC, Metals and Inorganic Groups. Much of this information can be found on the LEVEL 2 CHECKLIST's. For example, in metals analysis using graphite furnace analysis, the analysts and supervisor will examine instrument standardization criteria (absorbance for standards,etc.), dilution factors, linear range compliance, detection limit adjustment and whether the Method of Standard Additions was required.

SUPERVISOR:

It is recognized that the analyst supervisors are not a part of the data review group. However, the supervisors are directly responsible for the analytical performance of the various analyst and, as such, are an integral part of the review process. The main functions of the supervisors are to review analysis as soon as possible and 1) accept analysis or 2) suggest re-analysis. As part of

the LEVEL 1 REVIEW process, supervisors will perform the following tasks:

1. Review analysis package for QC, reasonable results, holding tie violations and general acceptance of analysis. It is very important that re-analysis decisions be made at this level.

2. Signify approval on LEVEL 1 REVIEW CHECKLIST

3. Approval of data entry into data base management system

4. Schedule data entry (applicable to inorganics analysis at this time only).

It will be the responsibility of the supervisor to review and approve (or disapprove) the analysis on a daily basis. It will not be acceptable for supervisors to allow their review packages to stack up while other tasks are being performed. The review process depends on a continual flow of information through the the various levels. To meet turnaround times and other constraints of a commercial laboratory, it is essential for supervisors to provide a timely review of data generated by the analysts.

B. LEVEL 2 REVIEW: DATA REVIEW GROUPS

At the present time, separate data review groups exist in the inorganics division, the GC/MS division and the Chromatography division. A thorough review of the project data base takes place within the data review groups. There are numerous items which are common to each divisions review procedure. Each review group has developed a separate checklist to aid each reviewer in specifics related to the analytical tests. In addition, the reviewers in each group possess sufficient experience with the analyses conducted by the division to allow a comprehensive assessment of the precision and accuracy of the data generated.

The LEVEL 2 REVIEW is considered to be a peer review of the analytical data and review of project specific requirements. At this stage of the review, a complete check of the tests assigned to a project is made against the project data base to assess project completion. Additionally, the preparation lab notes, bench sheets, QC forms and anomally sheets are reviewed in detail to ensure that raw data has been interpreted correctly, that detection, precision and accuracy criteria are met, that the information is well documented for archival purposes, and that contractual requirements are also met.

Each data review group will evaluate the project data with respect to the LEVEL 2 REVIEW checklists. If any re-analysis is required at this stage, the decision is documented along with other project specific data. The LEVEL 2 REVIEW CHECKLISTS for each group are shown in Figures 3-5. The completion of the LEVEL 2 REVIEW is indicated on the checklists by the appropriate signature.

The reviewers will also provide information which is used by the report preparation personnel to prepare the final project report. Reviewers should provide comments on unusual or inconsistent results, anomalies, subcontractor data, and the extent of any necessary data qualification. Reviewers are to also assemble the complete package for report generation, including the above comments and raw data, when requested.

Following the completion of the review by the peer reviewers, the complete package will be examined by the data review supervisor. Supervisors will provide additional review of comments, anomalies, data qualification, and relationships between parameters, when appropriate. Approval of the LEVEL 2 REVIEW by the supervisor is also indicated on the LEVEL 2 REVIEW CHECKLIST.

The supervisors will also check the file for completeness, address comments from reviewers, and spot check results for reasonableness. The supervisors will also develop revisions to the data review SOP, provide training to data reviewers, assist development of computer knowledge-based review software and provide a continued evaluation of data review procedures.

At the completion of the review process for each division, the supervisor will change the project completion status in LIMS from status 4 to 7. Altering the project status in this way allows management to effectively move projects through the laboratory as rapidly as possible.

C: LEVEL 3 REVIEW: CLIENT MANAGERS

The last review of project data takes place at the client manager level. This review is directed at the results obtained, the clients needs, overall project results across analytical divisions, special instructions, analysis problems, the extent of data qualification. Client managers are not responsible for numerical errors, wrong analysis dates and other information which is the responsibility of LEVEL 1 and LEVEL 2 REVIEW.

5. Responsibilities

LEVEL 1 REVIEW

The operations supervisors are directly responsible for the approval of the analysis and the LEVEL 1 REVIEW CHECKLIST. The analysts are responsible for the analyst items on the checklist and being aware of what takes place during LEVEL 2 REVIEW.

LEVEL 2 REVIEW

The peer reviewers in each data review group (inorg., GC/MS and chromatography) are responsible for the detailed review of all project information as indicated on the LEVEL 2 REVIEW CHECKLIST. The data review supervisor is responsible for a brief examination of the project data and comments, additional comments appropriate for the final report, training reviewers, and developing review procedures to be used for the LEVEL 1 and LEVEL 2 REVIEW.

LEVEL 3 REVIEW

The client managers are responsible for ensuring that the client's needs have been met, that the data appears reasonable and that contractual requirements have been met.

6. Comments

For the review process to be effective in correcting problems and improving data generated in the laboratory, it is essential that reviewers inform operations supervisors and client managers on a regular basis of the problems which have been identified during the review process. Review checklists or written memos would be an effective means for alerting various personnel on problems which could be avoided or should be corrected.

LABORATORY DATA REVIEW FRAMEWORK

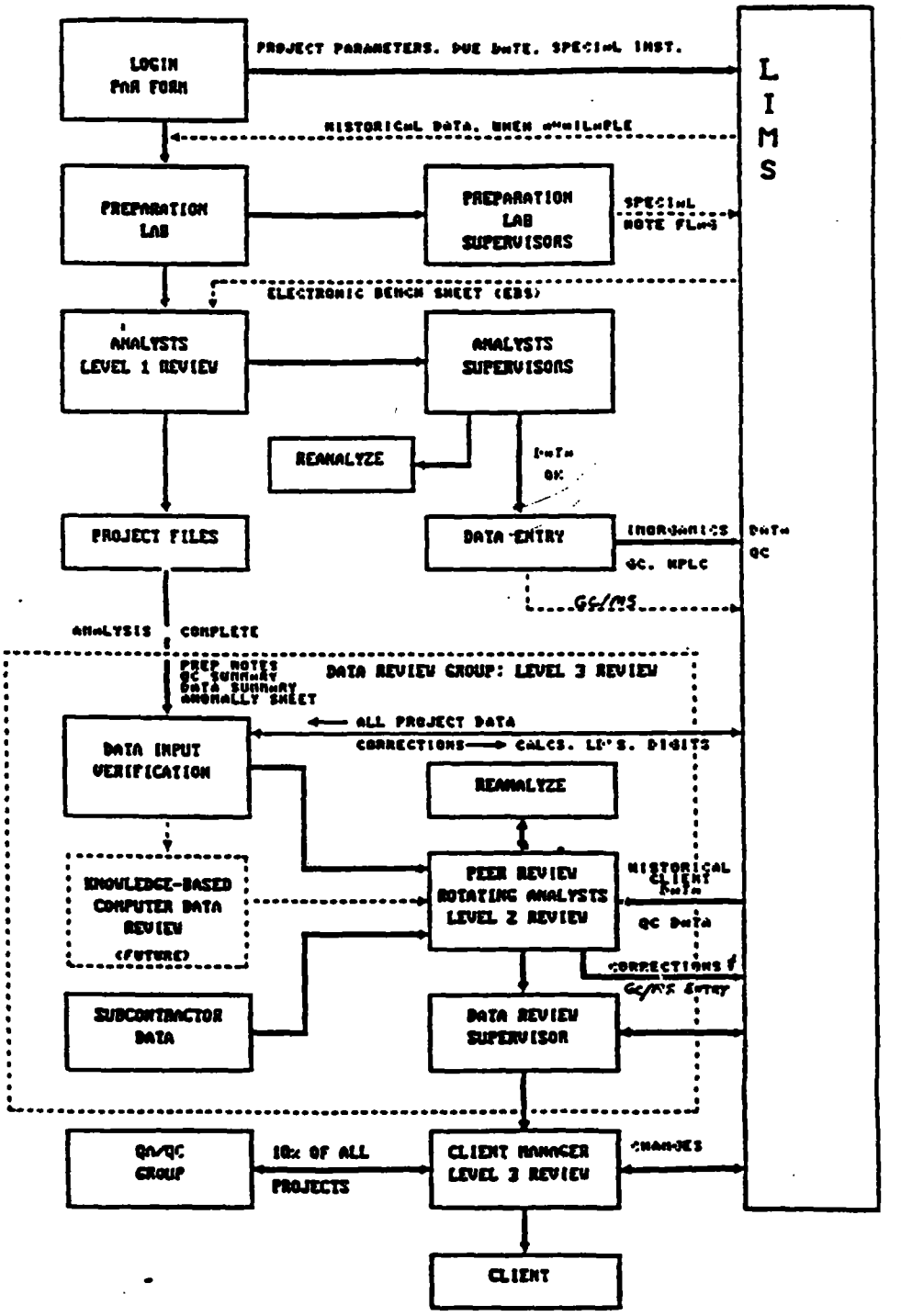


Figure 1 - Laboratory Data Review Framework Form

DATA REVIEW PROGRAM

LEVEL 1 REVIEW CHECKLIST PROJECT # _____

ANALYTICAL TEST_____

ANALYST ITEMS

Y N NA Preparation Lab Notes Reviewed

Y N NA Special Instructions Followed

Y N NA Samples Properly Preserved and in Proper Container

Y N NA Bench Sheets (Data Package Completed With All Information, Including Special Instructions

Y N NA Blank Correction Procedure Followed

Y N NA All Calculations Checked

Y N NA QC Within Limits

Y N NA Out of Control Form Filed

Y N NA Analysis Anomallies Noted

ANALYST COMMENTS:

ANALYSTS REVIEW_____ DATE _____

SUPERVISOR ITEMS

Y N NA Results Appear Reasonable

Y N NA Re-run Decision Documented

Y N NA Holding Time Violations Documented

SUPERVISOR COMMENTS:

SUPERVISOR APPROVAL_____ DATE _____

DATE DATA ENTERED _____BY WHOM_____

Figure 2 - Data Review Program Form

DATA REVIEW PROGRAM

LEVEL 2 REVIEW CHECKLIST PROJECT # _____

INORGANICS: METALS ____ NON-METALS ____

Y N NA Project Assignment Record (LIMS) vs. Actual Data

Y N NA Preparation Lab Notes Reviewed

Y N NA Special Instructions Followed, Check Item

____ Project Specific QC
____ Raw Data Requested
____ Limited Sample Volume
____ Special Preparation Needed
____ Custom Analytical Test
____ Special Holding Times
____ Other_____

Y N NA Bench Sheets (Analysis Package) Complete
Y N NA Special Instructions Noted
Y N NA Detection Limits Correct
Y N NA Blank Correction Procedure Followed
Y N NA Significant Digits Correct
Y N NA All Calculations Checked

Y N NA QC Checked and Acceptable
Y N NA QC Lot Assignment Correct
Y N NA Out of Control Form Filed

Y N NA Analysis Anomallies Noted

Y N NA Re-run Decision Documented

Y N NA Analysis Date Reflects Date of Accepted Data

Y N NA Holding Time Violations Documented

Y N NA Camera-Ready Report Cover Sheets Completed
Y N NA Prep sheet Attached
Y N NA Analysis Anomally Sheet Attached
Y N NA Raw Data Attached

LEVEL 2 REVIEW APPROVAL_____ DATE _____

CORRECTIONS ENTERED_____ DATE _____

SUPERVISOR APPROVAL_____ DATE _____

Figure 3 - Data Review Program Form

GC/MS DATA REVIEW CHECKLIST

- ☐ 1. Check LIMS Test vs SOP.
- ☐ 2. Check anomalies sheet and QC forms.
- ☐ 3. Check standard and see if it was updated.
- 4. Look at chromatogram for:
  - ☐ a. carry-over
  - ☐ b. truncating peaks
  - ☐ c. general chromatographic quality
  - ☐ d. very large unknown peaks
- ☐ 5. Recalculate run factors.
- ☐ 6. Check surrogates.
- 7. Check Quant list for:
  - ☐ a. linear ranges
  - ☐ b. co-eluting compounds
  - ☐ c. IS areas
  - ☐ d. carry-over
- ☐ 8. Check spectra for ID's and saturation.
- ☐ 9. Check if TID's are pulled if necessary.
- ☐ 10. Check chromatogram vs Quant list vs TID's.
- ☐ 11. Recalculate all target compounds and TID's.
- ☐ 12. Note any anomalies not on form already.
- ☐ 13. Over-all project review (compound types, ratios).

Figure 4 - GC/MS Data Review Checklist Form

DATA REVIEW PROGRAM

LEVEL 2 REVIEW CHECKLIST PROJECT # _____

CHROMATOGRAPHY

Y N NA Project Assignment Record (LIMS) vs. Actual Data

Y N NA Preparation Lab Notes Reviewed

Y N NA Special Instructions Followed, Check Item
- ____ Project Specific QC
- ____ Raw Data Requested
- ____ Limited Sample Volume
- ____ Special Preparation Needed
- ____ Custom Analytical Test
- ____ Special Holding Times
- ____ Other_____

Y N NA Bench Sheets (Analysis Package) Complete
Y N NA Special Instructions Noted
Y N NA Detection Limits Correct
Y N NA Blank Correction Procedure Followed
Y N NA Significant Digits Correct
Y N NA All Calculations Checked

Y N NA QC Checked and Acceptable
Y N NA QC Lot Assignment Correct
Y N NA Out of Control Form Filed

Y N NA Analysis Anomallies Noted

Y N NA Re-run Decision Documented

Y N NA Analysis Date Reflects Date of Accepted Data

Y N NA Holding Time Violations Documented

Y N NA Camera-Ready Report Cover Sheets Completed
Y N NA Prep sheet Attached
Y N NA Analysis Anomally Sheet Attached
Y N NA Raw Data Attached

LEVEL 2 REVIEW APPROVAL_____ DATE _____

CORRECTIONS ENTERED_____ DATE _____

SUPERVISOR APPROVAL_____ DATE _____

Figure 5 - Data Review Program Form

STANDARD
OPERATING
PROCEDURE

Subject or Title:
SAMPLE LOG-IN - REFER TO QAPP SECTION 7.1

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0003	Original	12/9/87

Supersedes:

1. Purpose:

To create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

Log-in must be completed within 24 hours of authorization by a project manager. Authorization occurs when a PAR, Menu of Analytical Services (Figure 1), and Project Screen in the computer are filled out and the information is turned in to the Receiving area (Client Services).

3. Safety: Not Applicable

4. Procedure:

As log-in proceeds fill out the LOG-IN checklist (Figure 2) and address all the issues on this form.

a. Retrieve the samples from the walk in cooler. Compare the bottles with the paperwork and PAR. Check for the correct test and sample matrix and properly preserved bottles for tests.
b. Check that the labeling was done correctly and referenced properly on the chain of custody.
c. Read the Menu of Analytical Services and PAR. Check for any jobcodes and special instructions. Special instructions are entered in the computer using a text editor and are then visible to all analysts. These are entered by the project managers.
d. Go to Data Set Maintenance in the Computer. Modify the project screen to change the project from P(planned) to A(active). Verify the number of samples and the amount of $.
e. Using the group code editor in LIMS (laboratory computer) set up groups of tests according to the PAR. Duplicate and Matrix spiked samples (Project specific QC) need to have separate groups of tests. Print copies of these group codes.

Prepared by: Robert K. Hall	Date: 12/10/87
Management Approval: Paul Webb	Date: 12/10/87
QA Officer Approval: Robert C. Hamisch	Date: 12/9/87

f. Go to the Log-in sample program. Assign the proper group code (groups of tests) to each sample separately, adding the received date, collections date, and client identification. List the bottles received for each sample and the location they will be placed. Samples placed on a hold status may or may not be logged in depending on the decision by the project manager.

g. Print a list of the samples, tests assigned, and bottles received through a select report. Print a copy of the project screen. Print an Acknowledgement letter indicating the samples received, RMAL numbers, and any discrepancies noted upon receipt.

h. Perform any compositing, filtering, or splitting necessary. Create any additional preserved bottles if necessary.

i. Fill out the Analysis request form for subcontract work (Figure 3) if necessary. A purchase order must also be filled out. For subcontracting to another Enseco facility fill out the Interlaboratory Analysis Request form (Figure 4).

j. Put samples in the proper locations. Volatiles are placed in refrigerators near the MS and GC areas. Waters for organic prep are placed in refrigerators near the Organic prep labs. Inorganic water bottles are placed in the walk in cooler and arranged by type of preservative. Solids and Wastes are stored in the walk in refrigerator on color coded shelves that are cross referenced by a color coded board in the receiving area.

k. All printed paperwork is placed in the project folder and it is reviewed by the supervisor. The folder is then stored in the Receiving area while the project is active.

l. Changes that need to be made to a project after log-in must be requested by filling out the Log in Change form (Figure 5).

5. Responsibilities:

Project manager is responsible for correctly filling out the PAR, special instructions, Menu of Analytical Services, and Project screen. Turnaround time is decided by the project manager. Sample Receiving technicians are responsible for transferring the information to the laboratory computer.

6. Definitions:

Special instructions - Typed instructions in LIMS to the operations groups and analysts that are necessary to complete the work and can not by indicated by using one of the computer tests.

Proj. #_____ Filled out by: _____

Client Name _____ Date: _____

MENU OF ANALYTICAL SERVICES (GENERAL INFO.)

Project manager must complete and submit this form to Sample Receiving along with PAR.

- **PROJECT TYPE:** [ ] Industrial [ ] EPA/CLP
- **WORK LEVEL DESIGNATION:** (specify one only) [ ] 1 [ ] 2 [ ] 3
- **ACKNOWLEDGMENT LETTER:** [ ] Normal [ ] Other Specify: _____
- **TURNAROUND:** [ ] Normal _____ (Report Due Date) [ ] Rush _____ (Verbal Results Due Date)

  _____ (Report Due Date)

  If rush, check boxes to indicate completion: [ ] Operation's Consent [ ] Rush mail message sent

  If rush, circle divisions involved: GC MS IN ME
- **BILLING:** List [ ] Std. Discount/Surcharge _____
  Note any special billing instructions:
  _____
  _____
  _____
- **SAMPLE DISPOSAL:**
  [ ] Return to client (30 days after completion of project.)
  [ ] Disposal by RMAL ($25/sample).
  [ ] Store beyond 30 days ($5/month/sample)
  Estimate additional time beyond 30 days. _____
  [ ] Other, specify:
  _____
  _____
- **REPORTABLES:**
  [ ] Standard RMAL "Camera Ready" Report only.
  [ ] Project Specific QC
  [ ] Special QC
  [ ] EPA/CLP, Specify package(s), (VOA, BNA, etc.)
  [ ] Special format
  [ ] Verbal results to client.

  Details:
  _____
  _____
  _____

Figure 1 - Menu of Analytical Services

LOG IN CHECKLIST

PROJECT #:_____ ADD ON #:_____

LOGGED BY:_____

PAR COMPLETE OR UPDATED?:_____

LIST PAR PROBLEMS:_____

_____

_____

PROJECT SCREEN CORRECT? (# of samples, $ amount)_____

SAMPLE MATRIX CORRECT?_____

TEST MATRIX CORRECT?_____

SHORT HOLDING TIMES ON BOARD?_____

VOAS ON BOARD?_____

CHAIN OF CUSTODY SIGNED?_____

SAMPLE BOTTLES LABELED CORRECTLY?_____

APPROPRIATE BOTTLES FOR PARAMETERS?_____

LIST BOTTLE PROBLEMS_____

_____

_____

VOA VIAL FOR VOA ANALYSIS?_____

SUBCONTRACT FORM FILLED OUT?_____

BOTTLES MADE TO SEND OUT?_____

BOTTLES BOXED AND PUT AWAY?_____

SPECIAL INSTRUCTIONS IN LIMS?_____

BNA OR VOA TESTS MODIFIED?_____

GROUP CODES SENT TO JEFF LOWRY?_____

Figure 2 - Log In Checklist

ANALYSIS REQUEST FOR SUBCONTRACTS

RMAL Project Number: _____

Laboratory to be subcontracted: _____

Project Manager: _____

Results Due: _____

Sample ID Number	Matrix	Parameter	Bottle Description	Comments

Figure 3 - Analysis Request for Subcontracts

INTERLABORATORY ANALYSIS

SHIP TO: *(circle one)*

CAL ERCO CLE GAS MAR HOU

Attention:

SEND RESULTS TO:

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611 FAC: (303) 431-7171

Attention:

CLIENT NAME | **PROJECT NO.**

Relinquished by: *(Signature)* **Received by:** *(Signature)* **Date** **Time**

Relinquished by: *(Signature)* **Received by:** *(Signature)* **Date** **Time**

Import Lab ID	Enseco ID	Client ID	Matrix (s, s, w)	Date Sampled	Date Rec'd	Date Auth.	Analysis Requested/ P.I. Item #	Sample Condition Upon Receipt

a. Written results required by (date): _____ Verbal results required by (date): _____

b. QC: ☐ Standard Enseco ☐ CLP Protocol ☐ Project-Specific _____

c. Sample Disposal: ☐ Enseco ☐ Return to Client ☐ Phone RMAL

d. Raw Data Copies Needed: ☐ Yes ☐ No

e. Detection Limits: ☐ Standard Product ☐ Other*

f. Holding Times: ☐ Enseco ☐ EPA-CLP ☐ Other*

g. *Special Instructions: _____

h. Intercompany Rebate: *(circle one)* 0% 5% 10% i. P.O. Number _____

LOG-IN CHANGES

DATE _____

PROJECT #_____

CHANGE REQUESTED BY _____

DESCRIBE CHANGE (tests added or deleted,corrections,etc.)

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

PRICE LIST INCREASE:_____

NEW DATA DUE:_____

NEW REPORT DUE:_____

PROJECT MANGER SIGNATURE_____

Figure 5 - Log In Changes

Enseco

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 29
USE OF PAR (Project Assignment Record) - Refer to QAPP Section 7.1

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0004	Original	12/9/87

Supersedes:

1. Purpose:

To designate and authorize the tests required for each sample (or sample site) and the matrix of these samples in order for a sample receipt technician to assign these tests in the lab computer.

2. Policies:

PAR's are always filled out before the log-in process may take place. Changing a standard list of analytes for a test logged in must be approved by a senior level manager.

3. Procedure:

a. Choose one of the 4 types of PARs.

Long Form - for projects involving Mass spec., Chromatography, Inorganic and metal work. (Figure 1).
Inorganic - for only inorganic and metals work. (Figure 2)
Chromatography - for only chromatography work. (Figure 3)
Mass Spectrometry - for only Mass spec. work. (Figure 4)

b. Fill in the information at top. Group the samples by similar tests required for the same sample matrix. Indicate the proper sample matrix (Figure 5). Indicate the proper test matrix (see the choices on the PAR).

For tests 01 - water
20 - solid
40 - waste
16 - TCLP
13 - EP TOX

Prepared by: [signature]	Date: 12/10/87
Management Approval: [signature]	Date: 12/10/87
QA Officer Approval: Robert C. Hamisch	Date: 12/9/87

c. Mark the columns associated with each group of samples for the test desired (with an x). Some exceptions are:

S or Q are required for some tests to indicate single or quad analyses

T or D are required to indicate Total or Dissolved

C is required to indicate a change to a standard list. For any C marked there must be an explanation written on the PAR. For example some analytes might be deleted or added from a standard Priority Pollutant Semivolatile list.

d. New tests that are not on the PAR must be created by the Data Administrator following completion of the Request form (Figure 6). Generic tests are available as place holders while the test is being created.

5. Responsibilities:

Project managers are responsible for the accuracy of the PAR.

6. Comments:

TCLP preps must be assigned. They are not pulled with the job codes. Some tests are not to be changed or modified (ICPLIT). Not all created tests are on the PAR. Most of the tests that RMAL sends to subcontractors must be hand written on the PAR. (Figure 7)

7. Definitions:

Jobcode - groups of tests that will be automatically assigned by the computer by the use of a simple phrase; example RCRA01C assigns all RCRA tests.

LONG FORM pg 1. *Last Revision: 6/8/87 Current Revision: 9/15/87* | Job Code Y N

Project #	Proj Mgr:	Prepared By:	Date: / /	Spcl Inst: Y N

GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

<<< >>> *<<<A >>>* **ORGANIC CHEMISTRY** *<<< >>>* *<<< >>>*

GC/LC Analyses	Test ID	Matrices	A	B	C	D	E
SDWA Trihalomethanes	THM SDW	01					
* Halogenated Volatile Organics **	601LI	01,20 ,16,46					
Halogenated VOA's (LOW DETECTION LIMIT)	601LIL	20					
* Aromatic Volatile Organics **	602LI	01,20 ,16,46					
Aromatic VOA's (LOW DETECTION LIMITS)	602LIL	20					
Benzene, Toluene, Ethylbenzene, Xylenes	602BTEX	01,20 ,16,46					
SDWA Volatiles	MCL SDW	01					
Acrolein & Acrylonitrile	*603LI*	*01,20*					
Phenols	604LI	01,20					
Benzidines	*605LI*	*01,20*					
Phthalate Esters	606LI	01,20					
Nitrosamines	607LI	01,20					
Organochlorine Pesticides/PCB's 608	OCP PP	01,20 ,16,46					
OC Pest's/PCB's (LOW DETECTION LIMIT)	OCP PPL	20					
NPDES Organochlorine Pesticides/PCB's	OCP PP	01,20					
* HSL Organochlorine Pesticides/PCB's **	OCP HSL	01,20					
HSL OCP's/PCB's (LOW DETECTION LIMIT)	OCPHSLL	20					
CLP/HSL Organochlorine Pesticides/PCB's	OCP CLP	01,20					
Appendix 8 or 9 Organochlorine Pest/PCB's	OCP AP9	01,20					
TCLP Characteristic Organochlorine Pests.	OCPTCLP	01,20,16,46					
* SDWA Organochlorine Pesticides	OCP SDW	01					
* RCRA Organochlorine Pesticides	OCP RCR	01,20					
PCB's	PCB	01,20, 45					
Nitro-Aromatics & Cyclic Ketones	*609LI*	*01,20*					
* Polynuclear Aromatic Hydrocarbons / 610	LC PNA	01,20 ,16,46					
Haloethers	*611LI*	*01,20*					
Chlorinated Hydrocarbons	*612LI*	*01,20*					
Organophosphate Pesticides **	OPP	01,20					

** Most Modifiable Test C - Change Noted
* Preferred Standard Product (01 & 20 Bolded) 01 & 20 Bolded - Std.Prd. DL

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 2. *Last Revision: 6/8/87* *Current Revision: 9/15/87*

GC/LC Analyses (cont.)	Test ID	Matrices	A	B	C	D	E
Appendix 8 or 9 Organophosphate Pesticides	OPP AP9	01,20					
Appendix 8 or 9 Herbicides	HRB AP9	01,20					
TCLP Herbicides	HRBTCLP	01,20, 16,46					
• SDWA Herbicides	HRB SDW	01					
* RCRA Herbicides **	HRB RCR	01,20					
Triazines	*619LI*	*01,20*					
Carbamate & Urea Pesticides/ HPLC	632LI	01,20					
Penta & Tetrachlorophenol	PCP	01,20					
Ethylene Dibromide (EDB)	504LI	01,20					
Hydrocarbon Scan by FID	GC HYD	01,20					
Boiling Point Distribution By GC	GC BPD	01,20					
Water Miscible Solvents	GC DAI	01					
Semivolatiles by MSD	MSD BNA	01,20					
Volatiles by MSD	MSD VOA	01,20					
Semivolatiles by FID	GC BNA	01,20					
Base Neutrals by FID	GC BN	01,20					
Acids by FID	GC ACD	01,20					
Land Treatment Demonstration / HPLC	LC LTD	01,20,43, 16,46					

GC / MS Analyses

Priority Pollutant Volatiles	VOA 624	01,20 ,40 16,46					
Pri. Pollutant VOA's (LOW DETECTION LIMIT)	VOA624L	20					
Priority Pollutant Semivolatiles	BNA 625	01,20 ,40 16,46					
Priority Pollutant Acid Organics	ACD 625	01,20 ,40					
Priority Pollutant Base/Neutral Organics	BN 625	01,20 ,40					
• Hazardous Substance List Volatiles	VOA HSL	01,20 ,40 16,46					
HSL Volatiles (LOW DETECTION LIMIT)	VOAHSLL	20					
• Hazardous Substance List Semivolatiles	BNA HSL	01,20,40 16,46					
* Hazardous Substance List Acid Organics	ACD HSL	01,20 ,40					
• Hazardous Substance List Base/Neutral Org	BN HSL	01,20,40					
CLP/HSL Volatiles (TID's Included)	VOA CLP	01,20,25					

** Most Modifiable Test C-Change noted. Bolded 01 & 20 - Std.Prd.

• Preferred Standard Product (01 & 20 Bolded)

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 3. *Last Revision: 6/8/87 Current Revision: 9/15/87*

GC / MS Analyses (cont.)	Test ID	Matrices	A	B	C	D	E
CLP/RSL Semivolatiles (TID's Included)	BNA CLP	01,20,25					
NPDES Volatiles Organics	VOA 624	01					
NPDES Semivolatiles Organics	BNA 625	01					
Appendix 8 or 9 Volatiles	VOA AP9	01,20,40, 16,46					
Appendix 8 or 9 Semivolatiles	BNA AP9	01,20,40, 16,46					
Appendix 8 or 9 Chlorinated Dioxins & Furns	DXN AP9	01,20 *Div.22*					
CL4-CL8 Dioxions & Furans	DXN	01,20 *Div.22*					
Appendix 8 TID Volatiles	VOATID8	01,20,40, 16,46					
Appendix 8 TID Semivolatiles	BNATID8	01,20,40, 16,46					
TCLP / Waste Characteristic Volatiles	VOATCLP	01,20,40, 16,46					
TCLP / Waste Characteristic Semivolatiles	BNATCLP	01,20,40, 16,46					
TCLP / Land Restriction Volatiles	VOA LRR	01,20,40, 16,46					
TCLP / Land Restriction Semivolatiles	BNA LRR	01,20,40, 16,46					
Refinery Hazardous Constituents Volatiles	VOA REF	01,20,40, 16,46					
Refinery VOA's (LOW DETECTION LIMIT)	VOAREFL	20					
Refinery Hazardous Constituents BNA	BNA REF	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons	BN PNA	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons SIM	BNSIPNA	01,20,40, 16,46					
Tentative Identification Volatiles	VOA TID	01,20,40, 16,46					
Tentative Identification Semivolatiles	BNA TID	01,20,40, 16,46					
Characterization Volatiles	VOA CHR	01,20,40, 16,46					
Characterization Semivolatiles	BNA CHR	01,20,40, 16,46					
Direct Aqueous Injection Volatiles	VOA DAI	01					
Direct Aqueous Injection Semivolatiles	BNA DAI	01					

Bolded 01 & 20 - Std. Prd.

C - Change Noted

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 4. *Last Revision: 6/8/87 Current Revision: 9/15/87*

<<< >>> <<< >>> INORGANIC CHEMISTRY <<< >>> <<< >>>

Physical Tests	Test ID	Matrices	A	B	C	D	E
@ Corrosivity By pH	METPH #	01,20					
Corrosivity, NACE	NACE	01,20					
@ Color	NESCOLR	01					
@ Odor	ODOR	01					
Particle Size / Hydrometer		20					
Particle Size / Sieve		20					
Ignitability, Closed Cup	FLSHPT	01,20,40					
Percent Oil/Water/Solid (O/W/S)	%OWS	40					
Percent O/W/S (Modified Oven Technique)	%OWSMOD	40					
Oil & Grease / Gravimetric	BAL O&G	01,20					
Oil & Grease / Infrared Spectrometer (IR)	IR O&G	01,20					
Aromatic Oil & Grease / (IR)	IR AO&G	01,20					
Total Petroleum Hydrocarbons (TPH) / (IR)	IR TPH	01,20					
Mineral Tests							
@ Specific Conductance	CELSC #	01,20					
@ Acidity	METACID	01,20					
@ pH	METPH #	01,20					
pH, Paste	METPHP	20					
@ Alkalinity, Total/Carb/Bicarb/Hydroxide	METALK	01,20					
Hardness, Titration	BURHARD						
Hardness, ICP	ICPHAR*						
Sodium Adsorption Ratio (SAR)	ICP SAR	01,20					
Cation Exchange Capacity	ICP CEC	20					
Ion Balance Calculation	IONBALCALC						
Ion Balance (Major Cations/Anions)	See Job Code: IONBALANCE						
Oxygen Demand / Carbon							
@ Biochemical Oxygen Demand (BOD)	METBOD	01					
Chemical Oxygen Demand (COD)	METCOD	01,20					
Total Organic Carbon (TOC)	TOCTOC#	01 *(20 Div.12)*					
Purgeable Organic Carbon (POC)	TOCPOC	01					
Dissolved Organic Carbon (DOC)	TOCDOC	01					

S-Single & Q-Quad for 01 Matrix Only; Other matrices do not need an additional letter
* T-Total, D-Dissolved, R-Recoverable.
@ SHORT HOLDING TIMES C-Change Noted.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 5. *Last Revision: 06/08/87 Current Revision: 09/15/87*

Nitrogen	Test ID	Matrices	A	B	C	D	E
Total Kjedahl Nitrogen (TKN)	TECTKN	01,20					
Ammonia, Nitrogen	TECNH3	01,20					
Ammonia, Distilled	TECNH3T	01,20					
@Nitrite, Nitrogen	TECNO2	01,20					
@Nitrate, Nitrogen	TECNO3	01,20					
Nitrite Plus Nitrate, Nitrogen	TECNOXT	01,20					
@ *Nitrate, IC*	*IC NO3*	*01,20*					
@ *Nitrite, IC*	*IC NO2*	*01,20*					
Total Organic Nitrogen	See Job Code : TON01						
Phosphorus							
@Orthophosphate, Colorimetric	TECO P	01,20					
@Orthophosphate, IC	IC PO4	01,20					
Polyphosphate, IC	IC PPO4	01,20					
Total Phosphorus, Colorimetric	TECT P	01,20					
Phosphorus, ICP	See ICP Suite Compounds						
Solids							
Total Solids (TS)	BALTS	01,20					
Total Suspended (TSS)	BALTSS	01					
@Total Dissolved Solids (TDS)	BALTDS	01					
Total Volatile Solids (TVS)	BALTVS	01,20					
Volatile Suspended Solids (VSS)	BALVSS	01					
@Turbidity (NTU)	SPETURB	01					
Settleable Solids (SS)	CONESS	01					
Microbiology							
@Coliform, Total	COLIF T	01					
@Coliform, Fecal	COLIF F	01					

* S-Single & Quad for 01 Matrix Only; Other matrices do not need an additional letter
* T-Total, D-Dissolved, R-Recoverable.
C-Change noted.

@ SHORT HOLDING TIMES Underlined Items Are Preferred

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 6. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Sulphur	Test ID	Matrices	A	B	C	D	E
Sulfate, IC	IC SO4	01,20					
Sulfate, Turbidimetric	SPESO4	01,20					
@ Sulfite, Titrimetric	BURSO3	01,20					
@ Sulfite, IC	IC SO3	01,20					
Sulfide, Colorimetric	SPES *	01,20					
Sulfide, IC	IC S	01,20					
Sulfur, ICP	See ICP Suite Compounds						
Sulfide - Reactive	SPES R	01,20					
Thiosulfate, IC	IC S2O3	01,20					
Thiocyanate, IC	IC SCN	01,20					
Cyanide							
Cyanide, Total	TECCN T	01,20					
Cyanide, Amenable to Chlorination	TECCN F	01,20					
Cyanide, Weak & Dissociable	TECCN W	01,20					
Cyanide, IC	IC CN	01,20					
Cyanide - Reactive	TECCN R	01,20					
Halogens							
Bromide, IC	IC BR	01,20					
Chloride, Titrimetric	BURCL	01,20					
Chloride, IC	IC CL	01,20					
@ Chlorine, Residual	POTCL2R	01,20					
Perchlorate, IC	IC ClO4	01,20					
Fluoride, Electrode	METF	01,20					
Fluoride, Distilled, Electrode	METF T	01,20					
Fluoride, IC	*IC F*	*01,20*					
Iodide, IC	IC I	01,20					
Total Organic Halogen (TOX)	TOXTOX#	01,20					
Purgeable Organic Halogen (POX)	TOXPOX	01					
Dissolved Organic Halogen (DOX)	TOXDOX	01					

S-Single & Q-Quad for 01 matrix only; Other matrices do not need an additional letter
* T-Total, D-Dissolved, R-Recoverable.
@ SHORT HOLDING TIMES, C-Change Noted. Underlined Items Are Preferred

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG-FORM. pg 7. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Radiochemistry	Test ID	Matrices	A	B	C	D	E
Gross Alpha & Beta	RADA&B	01,20					
Lead 210	RAPB210	01,20					
Radium 226	RAD226	01,20					
Radium 228	RAD228	01,20					
Thorium 230	RATH230	01,20					
Uranium, Natural	TORDOX	01,20					
Other Tests							
Tannin / Lignin		01,20 *Div.22*					
Phenolics (4-AAP)	SPEPHEN	01,20					
@ Surfactants (MBAS)	SPEMBAS	01,20					
CLIENTHOLD							
TCLP Master Preps							
TCLP Prep / EXTRACTABLE Organics Only**	M40TCLPO	40					
TCLP Prep / VOLATILE Organics Only	M40ZHE	40					
TCLP Prep / METALS Only	M40TCLPM	40					
TCLP Prep / METALS & EXRACT. ORGS Only	M40TCLP	40					

** Includes Pesticides

S-Single & Q-Quad for 01 Matrix Only; Other matrices do not need an additional letter.
* D-Dissolved, T-Total, R-Recoverable;
@ SHORT HOLDING TIMES C-Change Noted.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 8. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Trace Metals by ICP & AA	Test ID	Matrices	A	B	C	D	E
* ICP Scan / 27 Metals, Standard Product	ICP LI*	01,20					
ICP Metals, Soluble Salts	ICP SS	01,20					
ICP Suite / Choose From List Below	ICP*	01,20,T16,T46	See below				

Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E	Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E
Aluminum, ICP							Manganese, ICP						
Antimony, Furn AA	FSB*						Mercury, CV AA	CVHG*					
Antimony, ICP							Molybdenum, ICP						
Arsenic, Furn AA	FAS*						Nickel, ICP						
Arsenic, Hyd Gen	DIV. 22						Osmium, ICP						
Arsenic, ICP							Phosphorus, ICP						
Barium, ICP							Potassium, ICP						
Beryllium, ICP							Selenium, ICP						
Boron, ICP							Selenium, Furn AA	FSE*					
Cadmium, Furn AA	FCD*						Selenium, Hyd Gen	*Div. 22*					
Cadmium, ICP							Silica (SiO2), ICP						
Calcium, ICP							Silicon, ICP						
Chromium (III)	CR+3♦						Silver, Furn AA	FAG*					
Chromium (VI)	SPECR6*						Silver, ICP						
Chromium, ICP							Sodium, ICP						
Cobalt, ICP							Strontium, ICP						
Copper, ICP							Sulphur						
Iron, ICP							Thallium, Furn	FTL*					
Lead, Tot Organic							Tin, ICP						
Lead, Furnace AA	FPB*						Titanium, ICP						
Lead, ICP							Uranium, Natural						
Lithium, ICP							Vanadium, ICP						
Magnesium, ICP							Zinc, ICP						

* D-Dissolved, T-Total, R-Recoverable; (01,20,16,46 matrices for ICP* & Furnace Tests)
♦ DIS - Dissolved, TOT- Total.

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

LONG FORM pg 9. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Inorganic Regulatory Packages	Job Code	Matrices	A	B	C	D	E
Appendix VIII Metals/Inorganics	AP8**MI	01,09,20,40,16					
Appendix IX Metals/Inorganics	AP9**MI	01,09,20,40,16					
Appendix IX Optional-Water Chem. Parameters	IONBALANCE	09					
Hazardous Substance List (HSL) Met/Inorg	HSLA**MI	01,09,20,40,16					
CLP / HSL Metals/Inorganics	CLP**MI	01,09,20,40					
SDWA Primary Metals / Inorganics	SDWAP**M/I	01,09					
SDWA Secondary Metals / Inorganics	SDWAS**MI	01,09					
RCRA Total Metals	RCRA**M	01,09,20,40					
RCRA EP I Metals	EPI RCRM	Std.Prd. DL					
RCRA EP II Metals	EPII RCRM						
RCRA Groundwater Suitability	RCRAS**M/I/R	01,09					
RCRA Water Quality Metals/Inorganics	RCRAQ**MI	01,09					
RCRA Groundwater Quality Indicators	RCRAI**MI§	01,09					
Priority Pollutant Metals	PP**M	01,09,20,40,16					
Priority Pollutant Inorganics	PP**I	01,09,20,40					
Refinery Total Metals (Hazardous Constituent)	REFHC**M	01,09,20,40,16					
Refinery EP I Metals	EPI REFM	40					
Refinery EP II Metals	EPII REFM	40					
NPDES Part A Inorganics	NPDA**I	01					
NPDES Part B Metals / Inorganics / RAD	NPDB**MIR	01					
NPDES Part C Metals / Inorganics	PP**M/I	01,09,20,40					
TCLP Metals Aqueous Leachate	OTC**M	01,20,16					
TCLP Refinery Metals	See REFHC16M						

* S-Single, Q-Quad

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

S LONG FORM pg10. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Item Number	Changes

Comments To Sample Receiving :

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

JOB CODES pg 1 *Last. Revision: 4/2/87 Issued: 6/8/87*

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

JOB CODES pg 2. *Last Revision: 4/2/87 Issued: 6/8/87*

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCLP Waste Characteristic *(mark one: M/O/C)*	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCLP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!
YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCLP Prep / EXTRACTABLE Organics Only *	M40TCLPO	40						
TCLP Prep / VOLATILE Organics Only	M40ZHE	40						
TCLP Prep / METALS Only	M40TCLPM	40						
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40						

* Includes Pesticides

Figure 1 - Long Form for GC/MS, Inorganic, Metal Analyses

INORG CHEMISTRY pg 1. *Last Revision: 6/8/87 Current Revision: 9/15/87* Job Code : Y N

Project #	Proj. Mgr.:	Prepared By:	Date: / /	Spcl. Inst: Y N

Group	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

Physical Tests	Test ID	Matrices	A	B	C	D	E
@ Corrosivity by pH	METPH #	01,20					
Corrosivity, NACE	NACE	01,20					
@ Color	MESCOLR	01					
@ Odor	ODOR	01					
Particle Size / Hydrometer		20					
Particle Size / Sieve		20					
Ignitability, Closed Cup	FLSHPT	01,20,40					
Percent Oil / Water / Solids	%OWS	40					
Oil & Grease / Gravimetric	BAL O&G	01,20					
Oil & Grease / Infrared Spectrometer (IR)	IR O&G	01,20					
Aromatic Oil & Grease / IR	IR AO&G	01,20					
Total Petroleum Hydrocarbons (TPH) / IR	IR TPH	01,20					
Mineral Tests							
@ Specific Conductance	CELSC #	01,20					
@ Acidity	METACID	01,20					
@ pH	METPH #	01,20					
pH, Paste	METPHP	20					
@ Alkalinity, Total/Carb/Bicarb/Hydroxide	METALK	01,20					
Hardness, Titration	BURHARD						
Hardness, ICP	ICPHAR*						
Sodium Adsorption Ratio (SAR)	ICP SAR	01,20					
Cation Exchange Capacity	ICP CEC	20					
Ion Balance Calculation	IONBALCALC						
Ion Balance (Major Cations/Anions)	See Job Code: IONBALANCE						
Oxygen Demand / Carbon							
@ Biochemical Oxygen Demand (BOD)	METBOD	01					
Chemical Oxygen Demand (COD)	METCOD	01,20					
Total Organic Carbon (TOC)	TOCTOC#	01,*(20 Div.12)*					
Purgeable Organic Carbon (POC)	TOCPOC	01					
Dissolved Organic Carbon (DOC)	TOCDOC	01					

S-Single & Quad for 01 Matrix Only; Other matrices do not need an additional letter
** T-Total, D-Dissolved, R-Recoverable.
C-Change noted.

@ SHORT HOLDING TIMES

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 2. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Nitrogen	Test ID	Matrices	A	B	C	D	E
Total Kjedahl Nitrogen (TKN)	TECTKN	01,20					
Ammonia, Nitrogen	TECNH3	01,20					
Ammonia, Distilled	TECNHDT	01,20					
@ Nitrite, Nitrogen	TECNO2	01,20					
@ Nitrate, Nitrogen	TECNO3	01,20					
Nitrite Plus Nitrate, Nitrogen	TECNOXT	01,20					
@ *Nitrate, IC*	*IC NO3*	*01,20*					
@ *Nitrite, IC*	*IC NO2*	*01,20*					
Total Organic Nitrogen	See Job Code: TON01						
Phosphorus							
@ Orthophosphate, Colorimetric	TECO P	01,20					
@ Orthophosphate, IC	IC PO4	01,20					
Polyphosphate, IC	IC PPO4	01,20					
Total Phosphorus, Colorimetric	TECT P	01,20					
Phosphorus, ICP	See ICP Suite Compounds						
Solids							
Total Solids (TS)	BALTS	01,20					
Total Suspended Solids (TSS)	BALTSS	01					
Total Dissolved Solids (TDS)	BALTDS	01					
Total Volatile Solids (TVS)	BALTVS	01,20					
Volatile Suspended Solids (VSS)	BALVSS	01					
@ Turbidity (NTU)	SPETURB	01					
Settleable Solids (SS)	CONESS	01					
Microbiology							
@ Coliform, Total	COLIF T	01					
@ Coliform, Fecal	COLIF F	01					
Sulphur							
Sulfate, IC	IC SO4	01,20					
Sulfate, Turbidimetric	SPESO4	01,20					
@ Sulfite, Titrimetric	BURSO3	01,20					
@ Sulfite, IC	IC SO3	01,20					
Sulfide, Colorimetric	SPES *	01,20					
Sulfur, ICP	See ICP Suite Compounds						
Sulfide - Reactive	SPES R	01,20					
Thiosulfate, IC	IC S2O3	01,20					
Thiocyanate, IC	IC SCN	01,20					

* T-Total, D-Dissolved; for 01 matrix;
Total sulfide only, for 20 matrix.

Underlined Items Are Preferred

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 3. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Cyanide	Test ID	Matrices	A	B	C	D	E
Cyanide, Total	TECCN T	01,20					
Cyanide, Amenable to Chlorination	TECCN F	01,20					
Cyanide, Weak & Dissociable	TECCN W	01,20					
Cyanide, IC	IC CN	01,20					
Cyanide - Reactive	TECCN R	01,20					
Halogens							
Bromide, IC	IC BR	01,20					
Chloride, Titrimetric	BORCL	01,20					
Chloride, IC	IC CL	01,20					
@ Chlorine, Residual	POTCL2R	01,20					
Perchlorate, IC	IC CLO4	01,20					
Fluoride, Electrode	*METF*	*01,20*					
Fluoride, Distilled, Electrode	METF T	01,20					
Fluoride, IC	IC F	01,20					
Iodide , IC	IC I	01,20					
Total Organic Halogen (TOX)	TOXTOX#	01,20					
Purgeable Organic Halogen (POX)	TOXPOX	01					
Dissolved Organic Halogen (DOX)	TOXDOX	01					
Radiochemistry							
Gross Alpha & Beta	RADA&B	01,20 *Div.12*					
Lead 210	RAPB210	01,20 *Div.12*					
Radium 226	RAD226	01,20 *Div.12*					
Radium 228	RAD228	01,20 *Div.12*					
Thorium 230	RATH230	01,20 *Div.12*					
Uranium, Natural	RADU	01,20 *Div.12*					
Other Tests							
@ Tannin / Lignin	DIV. 22	01,20					
Phenolics (4-AAP)	SPEPHEN	01,20					
@ Surfactants (MBAS)	SPEMBAS	01,20					
Major Anion Scan by Ion Chromatography	IC SCAN	01,20					
TCLP Master Prep							
TCLP Prep / METALS Only	M40TCLPM	40					

S-Single, Q-Quad, for 01 matrix only; Underlined Items Are Preferred
20 matrix leave blank.

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 4. *Last Revision: 6/8/87* *Current Revision: 9/15/87*

Trace Metals by ICP & AA	Test ID	Matrices	A	B	C	D	E
ICP Scan / 27 Metals, Standard Product	ICP LI*	01,20,16					
ICP Metals , Soluble Salts	ICP SS	01,20					
ICP Suite / Choose from Lists Below	ICP*	01,20,T16,T46	See below				

Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E	Choose: ICP Suite, AA Metals	Test ID	A	B	C	D	E
Aluminum, ICP							Magnesse, ICP						
Antimony, Furn AA	FSB*						Mercury, CV AA	CVHG*					
Antimony, ICP							Molybdenum, ICP						
Arsenic, Furn AA	FAS*						Nickel, ICP						
Arsenic, Hyd Gen	DIV. 22						Osmium, ICP						
Arsenic, ICP							Phosphorus, ICP						
Barium, ICP							Potassium, ICP						
Beryllium, ICP							Selenium, ICP						
Boron, ICP							Selenium, Furn AA	FSE*					
Cadmium, Furn AA	FCD*						Selenium, Hyd Gen	DIV. 22					
Cadmium, ICP							Silica (SiO2), ICP						
Calcium, ICP							Silicon, ICP						
Chromium (III)	CR+3♦						Silver, Furn AA	FAG*					
Chromium (VI)	SPECR6*						Silver, ICP						
Chromium, ICP							Sodium, ICP						
Cobalt, ICP							Strontium, ICP						
Copper, ICP							Sulphur						
Iron, ICP							Thallium, Furn	FTL*					
Lead, Tot Organic							Tin, ICP						
Lead, Furnace AA	FPB*						Titanium, ICP						
Lead, ICP							Uranium, Natural						
Lithium, ICP							Vanadium, ICP						
Magnesium, ICP							Zinc, ICP						

* D-Dissolved, T-Total, R-Recoverable; (01,20,16,46 matrix for ICP* and Furnace Tests)
♦ DIS - Dissolved, TOT - Total.

Figure 2 - Inorganic and Metal Analyses

INORGANIC CHEMISTRY pg 5. *Last Revision: 6/8/87 Current Revision: 9/15/87*

Inorganic Regulatory Packages	Job Code	Matrices	A	B	C	D	E
Appendix VIII Metals / Inorganics	AP8**MI	01,09,20,40,16					
Appendix IX Metals / Inorganics	AP9**MI	01,09,20,40,16					
Appendix IX Optional-Water Chemistry Param	IONBALANCE	09					
Hazardous Substance List (HSL) Met / Inorg	HSL**MI	01,09,20,40,16					
CLP / HSL Metals / Inorganics	CLP**MI	01,09,20,40					
SDWA Primary Metals / Inorganics	SDWAP**M/I	01,09					
SDWA Secondary Metals / Inorganics	SDWAS**MI	01,09					
RCRA Total Metals	RCRA**M	01,09,20,40,16					
RCRA EP I Metals	EPI RCRM						
RCRA EP II Metals	EPII RCRM						
RCRA Groundwater Suitability	RCRAS**M/I/R	01,09					
RCRA Water Quality Metals/Inorganics	RCRAQ**MI	01,09					
RCRA Groundwater Indicators	RCRAI**MI#	01,09					
Priority Pollutant Metals	PP**M	01,09,20,40,16					
Priority Pollutant Inorganics	PP**I	01,09,20,40					
Refinery Total Metals (Hazardous Constituent)	REFHC**M	01,09,20,40,16					
Refinery EP I Metals	EPI REFM	40					
Refinery EP II Metals	EPII REFM	40					
NPDES Part A Inorganics	NPDA**I	01					
NPDES Part B Metals / Inorganics / RAD	NPDB**MIR	01					
NPDES Part C Metals / Inorganics	PP**M/I	01,09,20,40					
TCLP Metals Waste Characteristic Metals	OTC**M	01,20,16					
TCLP Refinery Metals	See REFHC**M						

Item Number	Changes

Comments For Sample Receiving:

S -Single, Q -Quad for 01 matrix only

Figure 2 - Inorganic and Metal Analyses

JOB CODES pg 1 *Last Revision: 4/2/87 Issued: 6/8/87*

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 2 - Inorganic and Metal Analyses

JOB CODES pg 2. *Last Revision: 4/2/87 Issued: 6/8/87*

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCLP Waste Characteristic *(mark one: M/O/C)*	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCLP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!
YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCLP Prep / EXTRACTABLE Organics Only *	M40TCLPO	40					
TCLP Prep / VOLATILE Organics Only	M40ZHE	40					
TCLP Prep / METALS Only	M40TCLPM	40					
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40					

* Includes Pesticides

Figure 2 - Inorganic and Metal Analyses

ORGANIC CHEMISTRY pg 1 *Last Revision: 4/2/87 Issued: 6/8/87* Job Code Y N

Project #	Proj Mgr:	Prepared By:	Date: / /	Spcl Inst: Y N

GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

GC/LC Analyses	Test ID	Approved Test Matrices	A	B	C	D	E
* SDWA Trihalomethanes	THM SDW	01					
* Halogenated Volatile Organics **	601LI	01,20,16,46					
Halogenated VOA Orgs (LOW DETECTION LIMIT)	601LIL	20					
* Aromatic Volatile Organics **	602LI	01,20,16,46					
Aromatic VOA Orgs (LOW DETECTION LIMIT)	602LIL	20					
Benzene, Toluene, Ethylbenzene, Xylenes	602BTEX	01,20,16,46					
SDWA Volatiles	MCL SDW	01					
Acrolein & Acrylonitrile	*603LI*	*01,20*					
Phenols	604LI	01,20					
Benzidines	*605LI*	*01,20*					
Phthalate Esters	606LI	01,20					
Nitrosamines	607LI	01,20					
Organochlorine Pesticides/PCB's 608	OCP PP	01,20					
OCP's/PCB's 608 (LOW DETECTION LIMIT)	OCP PPL	20					
NPDES Organochlorine Pesticides/PCB's	OCP PP	01,20					
* HSL Organochlorine Pesticides/PCB's **	OCP HSL	01,20,16,46					
HSL OCP's/PCB's (LOW DETECTION LIMIT)	OCPHSLL	20					
CLP/HSL Organochlorine Pesticides/PCB's	OCP CLP	01,20					
Appendix 8 or 9 Organochlorine Pest/PCB's	OCP AP9	01,20					
TCLP Characteristic Organochlorine Pests.	OCPTCLP	01,20,16,46					
* SDWA Organochlorine Pesticides	OCP SDW	01					
* RCRA Organochlorine Pesticides	OCP RCR	01,20					
PCB's	PCB	01,20,45					
Nitro-Aromatics & Cyclic Ketones	*609LI*	*01,20*					
Polynuclear Aromatic Hydrocarbons / 610	LC PNA	01,20,16,46					
Haloethers	*611LI*	*01,20*					
Chlorinated Hydrocarbons	*612LI*	*01,20*					
Organophosphate Pesticides **	OPP	01,20					
Appendix 8 or 9 Organophosphate Pesticides	OPP AP9	01,20					
Appendix 8 or 9 Herbicides	HRB AP9	01,20					

** Most Modifiable Test C - Change Noted
* Preferred Standard Product (Bolded 01 & 20) 01 & 20 Bol - Std.Prd. DL

Figure 3 - Chromotography Analyses

ORGANIC CHEMISTRY pg 2 *Last Revision: 4/2/87 Issued: 6/8/87*

GC/LC Analyses Cont.	Test ID	Matrices	A	B	C	D	E
TCLP Herbicides	HRBTCLP	01,20,16,46					
* SDWA Herbicides ##	HRB SDW	01					
* RCRA Herbicides ##	HRB RCR	01,20					
Triazines	*619LI*	*01,20*					
Carbamate & Urea Pesticides, HPLC	632LI	01,20					
Penta & Tetrachlorophenol	PCP	01,20					
Ethylene dibromide (EDB)	504LI	01,20					
Hydrocarbon Scan by FID	GC HYD	01,20					
Boiling Point Distribution By GC	GC BPD	01,20					
Water Miscible Solvents	GC DAI	01					
Semivolatiles by MSD	MSD BNA	01,20					
Volatiles by MSD	MSD VOA	01,20					
Semivolatiles by FID	GC BNA	01,20					
Base Neutrals by FID	GC BN	01,20					
Acids by FID	GC ACD	01,20					
Land Treatment Demonstration /HPLC	LC LTD	01,20,16,46					
CLIENTHOLD							

>>> Other <<<							
TCLP Prep / EXTRACTABLE ORGANICS Only	TCLPO	M40					
TCLP Prep / VOLATILES Only	ZHE	M40					

Item Number	Changes

Comments To Sample Receiving :

Most Modifiable Test

C- Change noted

* Preferred Standard Product (01 & 20 Bolded)

Figure 3 - Chromotography Analyses

JOB CODES pg 1 *Last Revision: 4/2/87 Issued: 6/8/87*

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I	01,09					
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40,16					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40,16					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40,16					
Organics	HSL**O	01,09,20,40,16					
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 3 - Chromotography Analyses

JOB CODES pg 2. *Last Revision: 4/2/87 Issued: 6/8/87*

Waste Characteristics & Other Tests	J.CODE ID	Matrices	A	B	C	D	E
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						
TCLP Waste Characteristic *(mark one: M/O/C)*	OTC**M/O/C	01,20,16					
Land Restriction Rule	LRR**O	01,20,16					
RCRA Metals	RCRA**M	01,09,20,40,16					
Refinery Hazardous Constituents (HC)							
HC Complete	REFHC**C	01,09,20,40,16					
HC Metals	REFHC**M	01,09,20,40,16					
HC Organics	REFHC**O	01,09,20,40,16					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40,16					
Metals / Inorganics	AP8**MI	01,09,20,40,16					
Organics	AP8**O	01,09,20,40,16					
Appendix 9 List							
Complete	AP9**C	01,09,20,40,16					
Metals / Inorganics	AP9**MI	01,09,20,40,16					
Organics	AP9**O	01,09,20,40,16					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					

TCLP MASTER,(M40), PREPS : THESE ARE NO LONGER INCLUDED IN JOB CODES !!
YOU MUST PIECE THE M PREPS TOGETHER WITH DESIRED ANALYSES ; SEE FLOWCHARTS

TCLP Prep / EXTRACTABLE Organics Only *	M40TCLPO	40						
TCLP Prep / VOLATILE Organics Only	M40ZHE	40						
TCLP Prep / METALS Only	M40TCLPM	40						
TCLP Prep / METALS & EXTRACT. ORGS Only	M40TCLP	40						

* Includes Pesticides

Figure 3 - Chromotography Analyses

ORGANIC CHEMISTRY pg 1 Issued: 01/07/87 Revised: 03/03/87 Job Code : Y N

Project #	Proj Mgr:	Prepared By:	Date: / /	Spcl Inst: Y N

GROUP	Smpl Mtx	Test Mtx	Client Description	RMA Sample Numbers
A				
B				
C				
D				
E				

Hazard Label:

GC / MS ANALYSES	Test ID	Approved Test Matrices	A	B	C	D	E
Priority Pollutant Volatiles	VOA 624	01,20,40 16,46					
Priority Pollutant Semivolatiles	BNA 625	01,20,40 16,46					
Priority Pollutant Acid Organics	ACD 625	01,20,40					
Priority Pollutant Base/Neutral Organics	BN 625	01,20,40					
* Hazardous Substance List Volatiles	VOA HSL	01,20,40 16,46					
* Hazardous Substance List Semivolatiles	BNA HSL	01,20,40 16,46					
Hazardous Substance List Acid Organics	ACD HSL	01,20,40					
Hazardous Substance List Base/Neutral Org	BN HSL	01,20,40					
CLP/HSL Volatiles (TID's Included)	VOA CLP	01,20					
CLP/HSL Semivolatiles (TID's Included)	BNA CLP	01,20					
NPDES Volatile Organics	VOA 624	01					
NPDES Semivolatile Organics	BNA 625	01					
Appendix 8 or 9 Volatiles	VOA AP9	01,20,40, 16,46					
Appendix 8 or 9 Semivolatiles	BNA AP9	01,20,40, 16,46					
Appendix 8 or 9 Chlorinated Dioxins & Furans	DXN AP9	Div. 22					
Appendix 8 TID Volatiles	VOATID8	01,20,40, 16,46					
Appendix 8 TID Semivolatiles	BNATID8	01,20,40, 16,46					
TCLP Waste Characteristic Volatiles	VOATCLP	01,20,40, 16,46					
TCLP / Waste Characteristic Semivolatiles	BNATCLP	01,20,40, 16,46					
TCLP / Land Restriction Volatiles	VOA LRR	01,20,40, 16,46					
TCLP / Land Restriction Semivolatiles	BNA LRR	01,20,40, 16,46					
Refinery Hazardous Constituents Volatiles	VOA REF	01,20,40, 16,46					
Refinery Hazardous Constituents BNA	BNA REF	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons	BN PNA	01,20,40, 16,46					
Polynuclear Aromatic Hydrocarbons SIM	BNSIPNA	01,20,40, 16,46					
Tentative Identification Volatiles	VOA TID	01,20,40, 16,46					
Tentative Identification Semivolatiles	BNA TID	01,20,40, 16,46					
Characterization Volatiles	VOA CHR	01,20,40, 16,46					
Characterization Semivolatiles	BNA CHR	01,20,40, 16,46					

C - Change Noted

* Preferred Standard Product (01 & 20 Bolded) Bolded 01 & 20 - Std. Prd.

Figure 4 - Mass Spectrometry Analyses

ORGANIC CHEMISTRY pg 2 Issued: 01/07/87 Revised: 03/03/87

GC / MS ANALYSES Cont.	Test ID	Matrices	A	B	C	D	E
Direct Aqueous Injection Volatiles	VOA DAI	01					
Direct Aqueous Injection Semivolatiles	BNA DAI	01					
>>> OTHER <<<							
TCLP Prep for Organics Only	TCLPO	M40					
TCLP Prep for Volatiles	ZHE	M40					
TCLP Prep for Organics & Metals	TCLP	M40					

Item Number	Changes

Comments To Sample Receiving :

Figure 4 - Mass Spectrometry Analyses

. JOB CODES pg 1. Issued: 03/03/87 Revised: 02/03/87

SDWA Drinking Water Parameters	J.CODE ID	Matrices	A	B	C	D	E
Primary Complete	SDWAP**C	01,09					
Primary Metals	SDWAP**M	01,09					
Primary Inorganics	SDWAP**I						
Primary Radiochemistry	SDWAP**R	01,09					
Primary Organics	SDWAP**O	01,09					
Secondary Metals / Inorganics	SDWAS**MI	01,09					
Priority Pollutants							
Complete	PP**C	01,09,20,40					
Metals	PP**M	01,09,20,40					
Inorganics	PP**I	01,09,20,40					
Organics	PP**O	01,09,20,40					
Hazardous Substance List							
Complete	HSL**C	01,09,20,40					
Metals/Inorganics	HSL**MI	01,09,20,40					
Organics	HSL**O	01,09,20,40					
CLP / EPA Report Packages							
Complete	CLP C						
Metals / Inorganics	CLP MI						
Organics	CLP O						
RCRA Groundwater - Monitoring Parameters							
Suitability Complete	RCRAS**C	01,09					
Suitability Metals	RCRAS**M	01,09					
Suitability Inorganics	RCRAS**I	01,09					
Suitability Radiochemistry	RCRAS**R	01,09					
Suitability Organics	RCRAS**O	01,09					
Quality Metals / Inorganics	RCRAQ**MI	01,09					
Indicator Inorganics	RCRAI**I#	01,09					
Waste Characteristics Tests							
WC Inorganics	RCRAWC40I	40					
EPI/ Metals	EPI RCRM						
EPI/ Organics	EPI RCRO						
EPII Oily Waste Metals	EPII RCRM						

S-Single, Q-Quad For 01 & 09 Matrices Only

Figure 4 - Mass Spectrometry Analyses

JOB CODES pg 2. Issued: 03/03/87 Revised: 02/0[illegible]/87

Refinery Hazardous Constituents (HC)	J.CODE ID	Matrices	A	B	C	D	E
HC Complete	REFHC**C	01,09,20,40					
HC Metals	REFHC**M	01,09,20,40					
HC Organics	REFHC**O	01,09,20,40					
Waste Characteristics - Refinery							
Inorganics	REFWC**I	40					
EPI Metals	EPI REFM						
EPII Oily Waste Metals	EPII REFM						
Appendix 8 List							
Complete	AP8**C	01,09,20,40					
Metals / Inorganics	AP8**MI	01,09,20,40					
Organics	AP8**O	01,09,20,40					
Appendix 9 List							
Complete	AP9**C	01,09,20,40					
Metals / Inorganics	AP9**MI	01,09,20,40					
Organics	AP9**O	01,09,20,40					
Ionbalance							
Complete	IONBALANCE	01,09					
Cations	CATIONS	01,09					
Anions	ANIONS	01,09					
TCLP - Refinery							
Complete	TCLPREF						
Metals	TCLPREFM						
Semivolatiles	TCLPREFBNA						
Volatiles	TCLPREFVOA						
TCLP - Waste Characteristics 6/13/86 Federal Registry							
(Federal Register) Complete	TCLPC						
Metals	TCLPM						
Semivolatiles	TCLPBNA						
Herbicides	TCLPHERB						
Pesticides	TCLPPEST						
TCLP - Land Restriction Rule							
Complete	TCLPLRRC						
TCLP Other							
601 List	TCLP601						
602 List	TCLP602						
PNA by 610	TCLPPNA						
Other							
RCRA Metals	RCRA**M	01,09,20,40					

Figure 4 - Mass Spectrometry Analyses

Enseco

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 7
SAMPLE RECEIPT AND CHAIN OF CUSTODY - REFER TO QAPP SECTION 7.1

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0005	Original	12/9/87

Supersedes:

1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is proceeded on them or not.

Project numbers are assigned in numerical order. USGS and MKE samples receive separate series of numbers. MKE samples require special chain of custody tracking.

3. Safety:

Always wear gloves and glasses while unpacking coolers. Coolers containing strong smelling samples must be unpacked under the hood area.

4. Procedure:

a. As samples arrive they are given a unique project number for each group of samples from one client and recorded in the log book (Figure 1).
b. Fill out the Sample checklist (Figure 2) while unpacking the samples.
c. For samples arriving by a courier check that the custody seals are intact.
d. Open the coolers, unpack the samples and check the information written on the chain of custody against what was received. Note any discrepancies such as missing samples, or broken bottles on the chain of custody form.
e. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications.

Prepared by: [illegible] Date: 12/10/87

Manageme pp val: Date: 12/10/87

A U c pp va : Date:

f. Sign and date the Chain of Custody (Figure 3). For samples hand delivered have the client sign and relinquish the custody. Always retain the top copy with the samples and only give a bottom copy to the client.
g. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding clipboard (Figure 4). Look for any volatile parameters and sign these samples in on the Volatile clipboard (Figure 5).
h. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, checklist, chain of custody and any paperwork received in the folder.
i. Deliver the file folder to the appropriate project manager.
j. Place the samples in boxes and store in the walk in cooler on special shelves pending log in. Bottles needed to analyze the short holding parameters are placed in a special location in the walk in cooler.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Client managers must notify sample receiving if others are to be included.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a special book and sign them in on return. The samples are stored in one of 3 locked refrigerators.

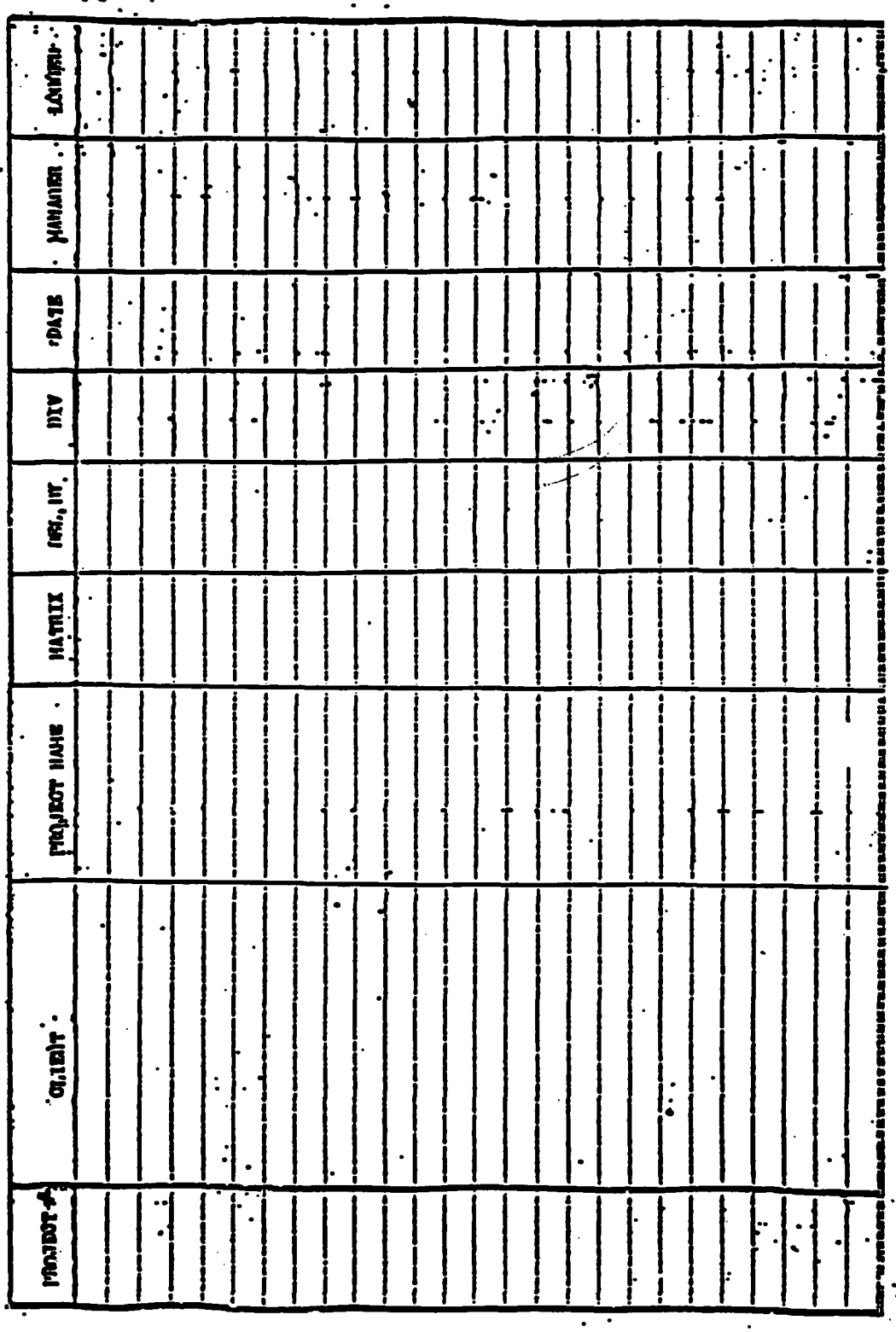


Figure 1 - Log Book Record

PROJECT # | | | | | | DIV: | | | | |

COMPANY NAME:_____
SAMPLE CHECKLIST DONE BY:_____

COOLER(S) #_____(RMAL/CLIENT)	Y	N
PICTURE TAKEN:		
SEALS INTACT:		
COOLER TEMP OK:		
BOTTLES BROKEN OR LEAKING:		
CONTAINERS LABELED:		
RADIATION DETECTION:		
CHAIN OF CUSTODY:		
CC AGREES WITH SAMPLES:		
VOA SAMPLES FILLED COMPLETLY:		
SEDIMENT PRESENT IN WATERS:		
SAMPLE CORRECTLY PRESERVED:		
SHORT HOLDING TIMES:		

( )MS ( )VOA ( )6OZ ( )IN

SAMPLE MATRIX: ( )WATER ( )SOIL ( )WASTE

OTHER:_____

TYPE OF BOTTLES: ( )RMA ( )CLIENT

DISCREPANCES:_____

Figure 2 - Sample Checklist

Enseco - Rocky Mountain Analytical

4955 Yarrow Street
Arvada, Colorado 80002
303/421-6611 Facsimile: 303/431-7171

Attn: _____

CHAIN OF CUSTODY

No.

Enseco Client _____

Project _____

Sampling Co. _____

Sampling Site _____

Team Leader _____

SAMPLE SAFE™ CONDITIONS

1. Packed by: _____ Seal # _____
2. Seal Intact Upon Receipt by Sampling Co.: Yes No
3. Condition of Contents: _____
4. Sealed for Shipping by: _____
5. Initial Contents Temp.: _____ °C Seal # _____
6. Sampling Status: Done Continuing Until _____
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab: _____ °C
9. Condition of Contents: _____

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by: (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: _____

Method of Shipment: _____ Airbill # _____

Received for Lab: _____ Signed: _____ Date/Time _____

Enseco Project No. _____

White and Pink Copies to Lab Yellow to Sampler

SS-001

Figure 3 - Sample Chain of Custody Record

INORGANIC SHORT HOLDING TIME PARAMETERS RECEIVED:_____ THIS PAGE COMPLETED:_____

SAMPLE NUMBERS:							
CHEM. DATE/TIME:							
PUT ON LIST BY/TIME:							
LABELS CHECKED:							
COLIFORMS (TOTAL)							
COLIFORMS (FECAL)							
Cr (VI)							
ALKALINITY							
24 HR pH (Q = Quad)							
48 HR CYAN (Q = Quad)							
RES. CHLORINE							
SULFITE							
OXOR							
BOD							
COLOR							
MBAS (SURFACTANTS)							
NITRATE-N							
48 HR NITRITE-N							
ORTHO-PHOSPHATE							
TURBIDITY							
SETTLEABLE SOLIDS							
IC SCAN							

SOP No. LP-RMA-0005
Page: 6 of 7
Date: 12/9/87
Revision: Original

Figure 4 - Inorganic Short Holding Clipboard

• MS/GC SHORT HOLDING TIMES.

DATE: _____

PROJECT NUMBER :							
COLL. DATE / TIME :							
REC. DATE / TIME :							
PROJECT MANAGER :							
MASS SPEC:							
HSL							
P. POLLUTANTS							
Refinery							
TCLP							
APPENDIX IX							
G. C. :							
BTEX							
602							
UNKNOWN	see manager	see manager	see manager	see manager	see manager	see manager	see manager
DONE BY							
COMMENTS:							

Figure 5 - Volatile Clipboard

PROCEDURES FOR GROUND WATER MONITORING:

Minnesota Pollution Control Agency Guidelines

April 1985

By

Gretchen V. Sabel
Pollution Control Specialist

Thomas P. Clark
Senior Hydrologist

This procedures manual was developed in July 1983 as one of the elements of the Minnesota Pollution Control Agency Ground Water Protection Strategy Work Plan, and revised following a lengthy comment and field trial period.

Major funding for this project was provided by the United States Environmental Protection Agency, Grant Number 005627011.

Minnesota Pollution Control Agency
Solid and Hazardous Waste Division
Program Development Section
612/296-7739

(REFER TO QAPP SECTION 6.5.4.)

ACKNOWLEDGMENTS

The authors wish to express thanks to those in the private sector as well as staff of other government agencies who provided valuable commentary on early drafts of this document. Thanks are also extended to all Minnesota Pollution Control Agency staff whose review and comment were greatly appreciated.

Special thanks go to the Program Development Section clerical staff for their typing of the many drafts and revisions of this document, and to Len Nelson, who drafted most of the diagrams and provided us with the cover art.

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

LIST OF FIGURES

control. Laboratory precision and accuracy data should be submitted to the Agency as a part of the quality assurance plan described in Section 9.

Error can be introduced at each stage of monitoring, including installation of monitoring devices, sampling from monitoring points, and analysis. It is the responsibility of all who are involved to keep this error to a minimum. Providing accurate and meaningful data the first time is to everyone's benefit.

Monitoring is not an end in itself. Rather it should be thought of as a tool used to measure a site design's efficiency at controlling water pollution. Continued monitoring is required after corrective measures have been taken to verify that water quality improves over time.

SECTION 2

MONITORING WELLS

Monitoring Well Construction

Construction of all water wells, including monitoring wells, is under the jurisdiction of the Minnesota Department of Health (MDH). The wells must be installed by either a licensed well driller or an engineer registered with the MDH to install monitoring wells. All applicable construction standards included in the Minnesota Water Well Construction Code must be met. The current standards are found in 7 MCAR §§ 1.210-1.224,* copies of which are available for a fee by writing:

Minnesota Department of
Administration
Documents Section
117 University Avenue
St. Paul, Minnesota 55155

The construction code is currently being amended to include separate specific requirements for ground water monitoring well construction. Figure 1 shows a well which meets current (March 1985) construction requirements.

Details of monitoring well construction will be determined by site-specific factors such as required depth of monitoring wells, waste types, and expected aquifer yield. For that reason, a detailed monitoring system plan showing geologic stratigraphy, well locations (both horizontally and vertically) and well construction details must be

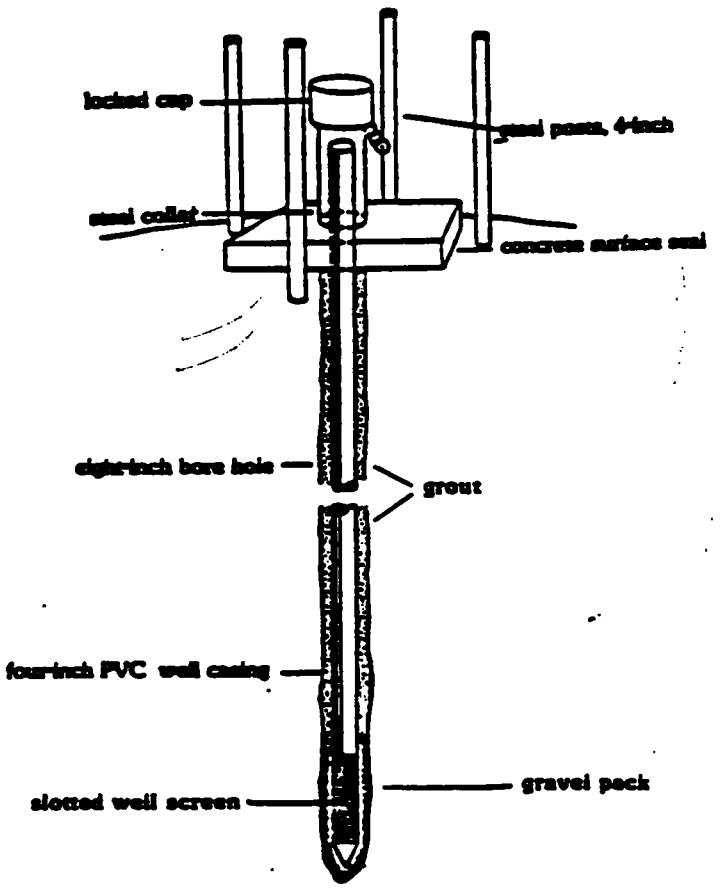


Figure 1. Monitoring Well Construction

*Subject to recodification in 1985, the Minnesota Water Well Construction Code will be found in Minnesota Rules Chapter 4725 (1985).

submitted to the Agency for approval prior to installation. Approval must also be obtained from the MDH before installation of wells which penetrate one or more confining beds. See "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985) for a more detailed discussion of monitoring network design.

Some specific requirements follow.

Well labeling:

Each well should be permanently labeled with its assigned monitoring point identification. The label should be consistent with the identification used in site plans and monitoring reports. One way to permanently label a well would be to inscribe the well identification number and the date of installation in the concrete slab surrounding the well collar while it is still wet. Alternately, the information may be etched or painted directly on the well collar.

Well diameters:

Wells must be constructed to facilitate sampling. Wells which are installed with water levels below the suction limit (approximately 25-30 feet) and which are too narrow in diameter to allow for sampling with a conventional submersible pump are unacceptable. Consideration should be given to the sampling method to be used when planning well construction. For wells less than 25 feet deep, a 2-inch diameter is sufficient because the wells may be sampled with a peristaltic pump. Two-inch wells, 25-125 feet deep, may be sampled with a 2-inch submersible pump, but these units are costly. In some instances, it may be more cost-effective to install 4-inch wells which can be sampled with less expensive, higher capacity submersible pumps. Wells greater than 125 feet deep should be at least 4 inches in diameter to allow for sampling with conventional submersible pumps. The Minnesota Water Well Construction Code requires a 2-inch annulus surrounding the well casing.

Casing material:

Well casings must meet MDH requirements, which currently allow only the use of PVC or ABS plastic, ferrous materials or stainless steel. A general rule of thumb is to choose threaded plastic pipe where metals and physical parameters are to be tested, ferrous pipe where organics alone are to be tested, and stainless steel where all types of parameters are to be tested. Since the casing material can cause interferences with some tests, it may be advisable to install 4-inch wells so that higher capacity pumps can be used to reduce casing influences on the sampled water quality. When PVC casing is installed, schedule 80 should be used to meet MDH strength requirements.

All materials used in well construction should be as clean as possible before installation. Washing with tap water is acceptable for general applications. More rigorous procedures, such as steam cleaning or hexane and distilled water rinses, are necessary when monitoring for very low levels of some contaminants. Care should be taken when using plastic pipe that solvent-welding compounds are not used, and that any cleaning fluids used are rinsed off with distilled water prior to installation. At least one screen and

casing manufacturer offers a cleaning service, providing the parts hermetically sealed in plastic bags for transport to the drill site.

Well screens and filter packs:

Well screen slots should be sized as large as possible to allow proper development, while preventing sediment from entering the well. Slot size should be based on grain-size distribution and hydrologic characteristics of the aquifer being monitored. Filter packs of washed sand or gravel are often necessary to produce sediment-free water for testing. Filter pack materials and grout should not be poured in from the surface. The Agency recommends these materials be placed from the bottom of the borehole using tremie pipes. Centering guides should be used on the screen and casing so the filter packs and grout can be placed uniformly around the well.

Grouting:

The annulus around the casing should be sealed with neat cement grout to prevent the flow of contaminated water along the casing. The use of bentonite is discouraged because its composition can vary widely, depending upon its source, which may subsequently affect the water quality analyses from that well. A small amount of bentonite (2%) may be added to the cement, however, to minimize shrinkage.

Well development:

Monitoring wells must be capable of producing nearly sediment-free water to provide meaningful analytical results. Thorough well development is therefore essential. The combined use of a jetting tool with air-lift pumping is a particularly effective development method. Mechanical surging as with a surge block or large bailer can also be used, but is less effective. Over-pumping is not recommended, as it may cause bridging of sand particles in the sand pack or formation.

Recent work suggests that wells in tight formations may be more effectively developed if the standing water is gently removed as it recharges the well, rather than agitating the walls of the annulus with the more aggressive development techniques. This benign development technique may take a longer period of time to successfully accomplish, but once complete can improve the yield of a well which otherwise would only provide marginal yield for sampling.

Well installation in areas of known or suspected contamination:

When working in areas of known or suspected contamination, or in any areas which may be affected by pollution sources, extreme care must be taken to avoid spreading contamination during the drilling operation. Drill stems and all down-hole equipment should be thoroughly steam-cleaned and, in some instances, hexane-rinsed to avoid spreading contaminants to uncontaminated areas of the site. Native soil should not be used as backfill. Clean sand should be brought in instead. Wells which penetrate a confining bed are required by MDH to be constructed with double casings.

Well drilling methods:

Wells may be drilled by a number of methods, a good discussion of which is given in "Manual of Ground-Water Sampling Procedures" (Scalf, 1981). The use of drilling fluids and muds is discouraged because of possible analytical interferences and well screen clogging.

Well drilling and construction logs:

High-quality drilling and laboratory identification logs must be submitted for each well or piezometer. These logs should include at least the following information:

1. Detailed soil or rock description and classification, measured soil and rock properties, sample collection locations and types of collection methods, and elevations of upper and lower contacts. Classification should be done by a qualified geologist or soils specialist. If the methods of drilling and sampling do not permit the soil characteristics and contact elevations to be described accurately and in detail, geophysical logs should be provided.

2. Location referenced to known control points, date of completion, and name, address, and telephone number of person or company responsible for construction of the well.

3. Depth of well; construction materials utilized, including well casing type and size, screen opening size; size fraction of screen packing material used for casing and grout; methods used to drill the hole, place the sealed intervals, and develop the well; elevations of the land surface, top of casing, and measured water levels; elevations of the top and bottom of the screen, casing, and each type of backfill or seal; intervals of screen packing material; grouted intervals; and geophysical logs, if any.

Stabilization and Recovery Rate Tests

Once a well is installed and developed, a pumping test of two to three hour duration may be conducted to obtain information on the well's yield potential and relative transmissivity (a measure of the rate at which water can be transmitted) of the aquifer. Recent research by the Illinois State Water Survey (Barcelona, 1983) recommends the short pumping test as a data-gathering method for determining "the frequency at which samples will be collected and the rate and period of time each well should be pumped prior to collecting the sample." Figure 2, from that reference, graphically shows the effect that aquifer transmissivity has on the amount of pumping needed to obtain a sample containing water derived mostly from the aquifer rather than water from storage within the casing and annular space. A more detailed discussion of this concept is given in Gibb, 1981.

While these pumping tests are not currently being required by the Agency on a routine basis, a stabilization test (or, alternately, a recovery rate test) is required for all new wells following development to determine the amount of pumping needed prior to sample collection. To conduct a stabilization test, pump the well to waste at a rate that yields a constant stream of water without

dewatering the well. This rate should be equivalent to the rate at which the well will be pumped during sampling. If the well has slow recharge and goes dry with pumping, a recovery rate test should be performed instead. The pump should be set in the upper portion of the water column so that all water standing in the well is drawn out and no stagnant water remains above the pump.

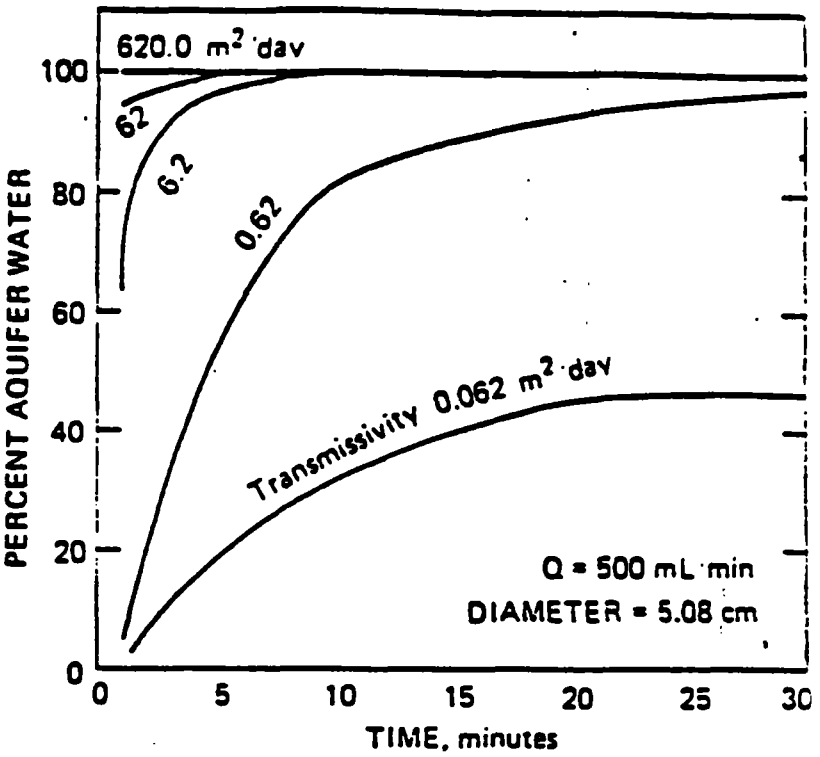


Figure 2. Relationship of Aquifer Transmissivity and Required Well Evacuation (From Barcelona, 1983)

Specific conductance, pH, and temperature should be measured in the field at intervals of one well volume until three successive readings yield equivalent values within the following ranges for each of these parameters:

Specific conductance (temperature-corrected): ± 10 umhos/cm
pH: ± 0.1 pH unit
Temperature: ± 0.5 °C

Once stabilized, samples should be collected without delay. In past versions of this manual, the measurements were to be taken at five minute intervals. Recent Agency experiences with monitoring wells have led to this change in instructions in an effort to avoid over-pumping the sampling wells. A closed, flow-through chamber is helpful for taking the measurements. The device shown in Figure 3 was fabricated by Dale Thompson of the MPCA for sampling at monitoring wells. For additional references on the stabilization test see U.S. Geological Survey, 1976; Gibb, 1981; and MPCA, 1983.

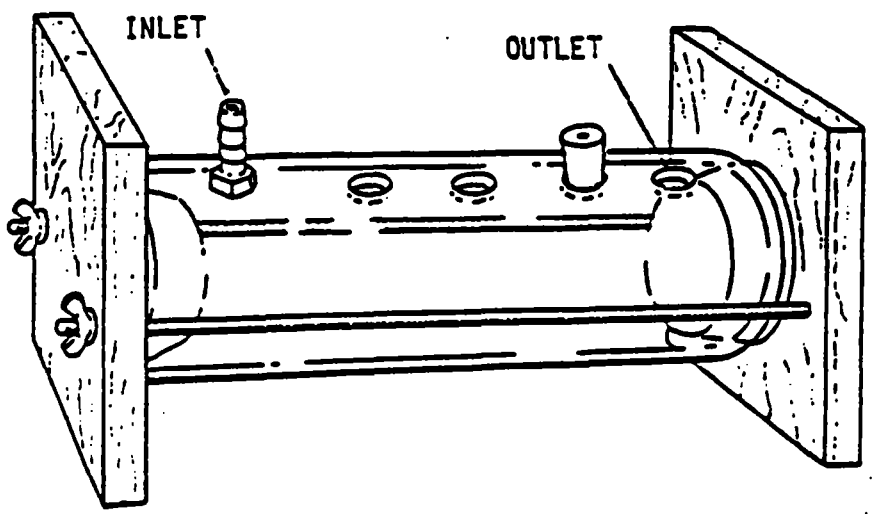


Figure 3. Flow-Through Stabilization Chamber

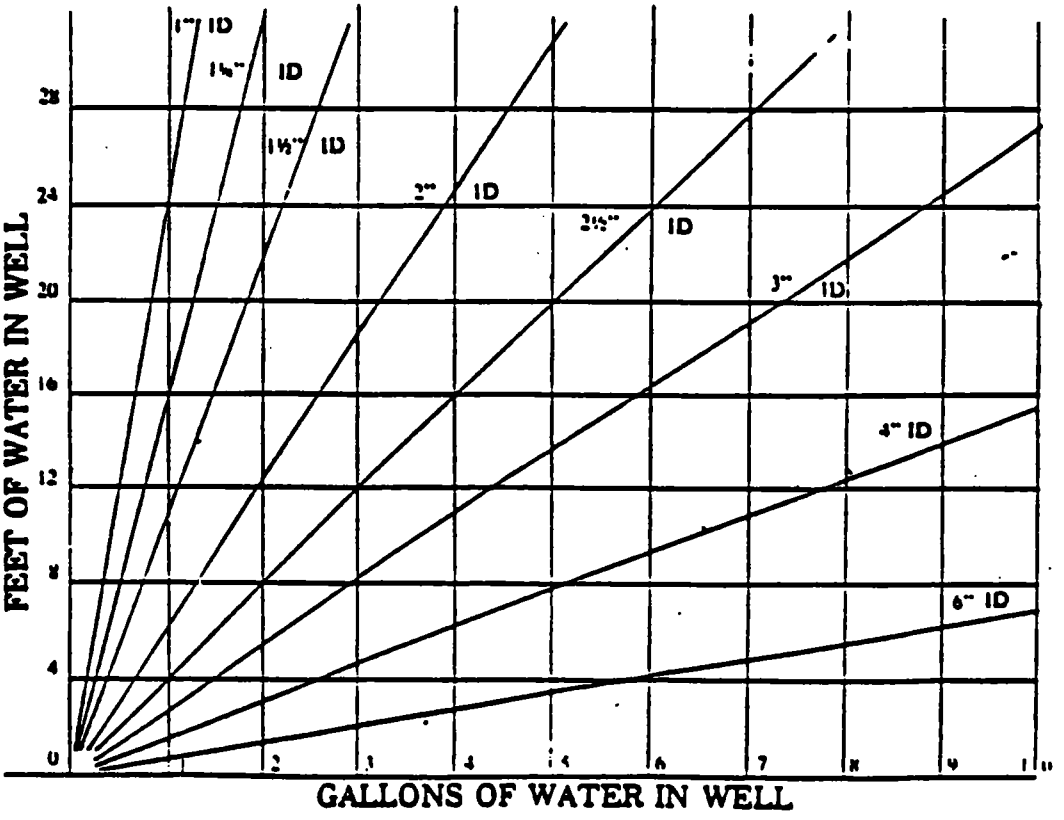
The results of the initial stabilization test should be recorded as suggested in Figure 4 and submitted to the Agency along with the well log and well construction details. If sampling methods change, the stabilization test should be repeated using the new equipment.

In some cases, it may be necessary to install wells in fairly tight (low transmissivity) formations which do not have sufficient yields to allow a stabilization test. For such wells, it is permissible to substitute a recovery rate test for the stabilization test. A recovery rate test is performed by taking initial measurements at the well of water elevation, pH, temperature and conductance; fully and as quickly as possible evacuating the well; measuring the water level as it recharges over time; and taking final measurements of water elevation, pH, temperature and conductance.

Figure 5 below provides a handy conversion for determining the volume of water in a well for various casing diameters.

Samples are collected as the well refills, not to exceed two hours after evacuation. For more information on interpreting results from a recovery rate test, see the section in Freeze, 1979 on the interpretation of slug test data. A suggested format for reporting the results is given in Figure 6.

Figure 5. Water in Storage



Well Diameter	Gallons Per Foot of Casing
1 1/4"	0.0625
2"	0.163
4"	0.653
	1.47
	2.61
12"	5.87

Figure 4

STABILIZATION TEST

Site _____

Date _____

Well number _____

Pumping rate (gallons/minute) _____

Type of pump _____

Water level before pumping (nearest 0.01 ft. below top of casing) _____

Approximate well location _____

Calculated volume of water in casing _____

Weather conditions _____

Time	pH (units)	Temperature-Corrected Conductance (umhos/cm)	Temperature (°C)	Water Level (nearest 0.01 ft.)	Cumulative Volume of Water Removed From Well (measured in gallons)

ENSR

WELL NO.

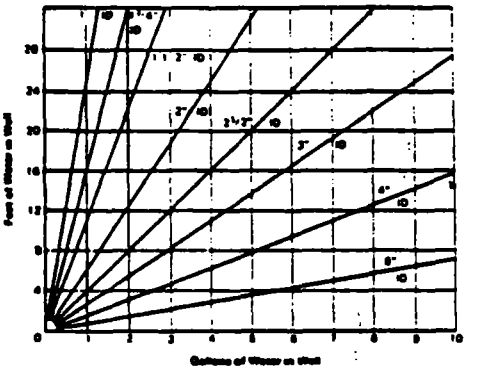
GROUND WATER SAMPLE COLLECTION RECORD

Project No. _____ Date _____ Time: Start _____ am/pm

Project Name _____ Finish _____ am/pm

Location _____

Weather Conds.: _____ Collector _____



1. WATER LEVEL DATA: (measured from ToC)

a. Total Well Length _____ Well Casing Type _____

b. Water Table Depth _____ Casing Diameter _____

c. Length of Water Column _____ (a-b)

d. Calculated Purgeable Volume _____

2. WELL PURGEABLE DATA

a. Purge Method _____

b. Required Purge Volume (@ _____ well volumes) _____

c. Field Testing: Equipment Used _____

Volume Removed	T°	PH	Spec. Cond.	Color	Other

3. SAMPLE COLLECTION: Method _____

Container Type	Preservation	Analysis Req.

Comments _____

M990244

Figure 5

RECOVERY RATE TEST

Site _____

Date _____

Well number _____

Water level before evacuation (nearest 0.1 ft. below top of casing) _____
Approximate well location _____
Weather conditions _____

Initial: pH (units) _____ Recharged: pH (units) _____
Conductance (umhos/cm*) _____ Conductance (umhos/cm*) _____
Temperature (°C) _____ Temperature (°C) _____

Time From Evacuation	Water Level (nr. 0.1 ft.)	Calculated Recharge Rate $(\frac{\text{volume}}{\text{time}})$

The test is finished when the water level has recovered to its pre-evacuation level.

*Conductance should be temperature-corrected to 25°C.

Monitoring Well Maintenance

A damaged or improperly constructed monitoring well can provide a conduit for contaminants to enter the ground water. In addition, monitoring data is most meaningful when it comes from repeated testing of the same well by the same methods under the same conditions. Therefore it is important that wells be maintained in good condition. Wells which are allowed to deteriorate and no longer provide suitable samples must be replaced.

Surface seals must be intact to prevent surface water entering the borehole. Obstructions in the well should be removed. Any well which cannot be sampled must be abandoned in accordance with the Minnesota Water Well Construction Code. If a well is lost, such as those accidentally buried by landfill equipment, excavations must be made to locate the well, so that it can be reconstructed or abandoned. Locking caps are necessary to prevent vandalism. Wells in vulnerable locations should be inspected weekly for damage.

Sample Collection from Monitoring Wells

Samples must be carefully and precisely collected. To assure that samples are taken correctly, appropriately trained laboratory personnel or consultants should collect the samples.

There is a wide selection of methods for collecting water samples from monitoring wells. Each method has advantages and disadvantages. The choice of method should be based upon well construction, the amount of water to be removed and the parameters of interest. Following is a discussion of several methods.

Squeeze or Bladder Pumps:

The major components of these systems include a collapsible bladder inside a long, rigid housing, a compressed gas supply and appropriate control valves. Water enters the bladder through the foot valve when the pump is submerged. Gas pressure is applied to the annular space between the rigid housing and the membrane, forcing the water up. The top check valve keeps the sample in the discharge line when the pressure is released, and more water enters the bladder through the foot valve.

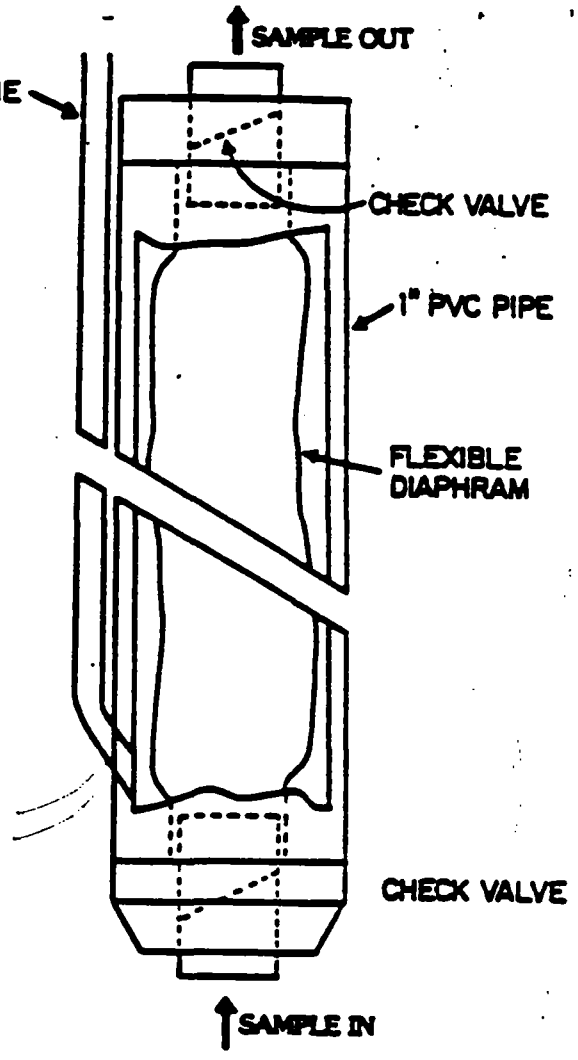


Figure 7. Squeeze or Bladder Pump

Advantages:

-wide range of pumping rates
-may be constructed of materials inert to the parameters of interest
-no gas-water contact; no out-gassing, stripping or loss of volatiles
-can be used well diameters as small as one inch
-portable, or may be used as a dedicated installation
-can be used to sample as deep as 200 feet

Disadvantages:

-large gas volumes and long cycles needed for deep operation
-pumping rate not as great as with submersible or suction pumps
-control unit is relatively expensive

Usage Recommendations:

-acceptable to use in most sampling applications
-pump should be set above the screen for evacuation and sampling
-attention must be given to adequate cleaning before each use to avoid cross-contamination of wells

Bailers:

Bailers can range in construction from a simple length of capped pipe on a rope to Teflon devices with a dual check-valve system used for sampling at discrete points in the water column. Wells are sampled by lowering the bailer to a given point in the water column and raising it, filled, to the surface.

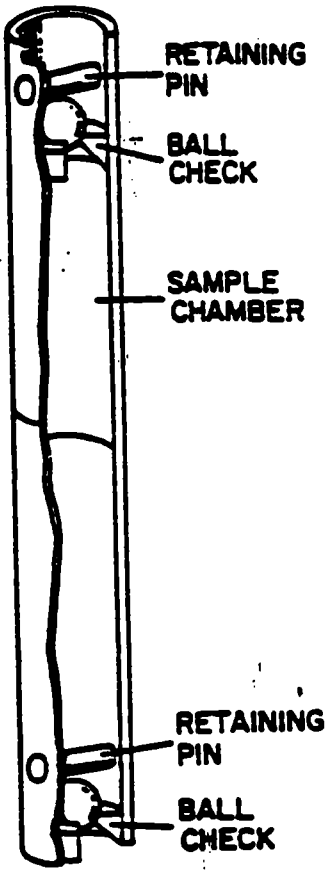


Figure 8. Timco Variable-Capacity Discrete Point Sampler

Advantages:

- -few, if any, mechanical difficulties encountered in use
- -very portable; economical to build from easily obtainable parts
- -construction materials can be chosen for compatibility with the parameters of interest
- -can be built to fit small diameter wells
- -low surface-to-volume ratio reduces loss of volatile components
- -can be easily laboratory cleaned and taken into the field sealed to keep clean

Disadvantages:

- -requires much manual labor if used for well evacuation
- -difficult to fill sample bottles without aeration of samples unless the bailer is equipped with a bottom emptying device
- -the large amount of handling of the bailer needed in sampling can lead to increased risk of sample contamination

Usage Recommendations: (from Gibb, 1981)

- -the bailer should be constructed of a noncontaminating material (such as stainless steel or Teflon)
- -a pass-through type valve should be used to minimize disturbance as the bailer is lowered through the water column
- -the bailer should be lowered to the same depth (the top of the well screen) every time to create the same effect as pumping with a peristatic pump
- -bailing should be timed to approach a constant pumping rate and should continue until the appropriate well volumes are removed prior to collecting the sample
- -the rope used to operate the bailer should be of a nylon, polyester, or Teflon-coated material and should be held off the ground during the bailing process
- -the bailer and down-hole rope should be thoroughly cleaned (at least rinsed with distilled water) before use in each well

Submersible Pumps:

A submersible pump consists of a stack of impellers, driven by a special motor designed for use under water. This type of pump is appropriately used in wells with large volumes of water, wells with large water columns and deep wells. Submersibles can be used as portable pumps or permanently installed.

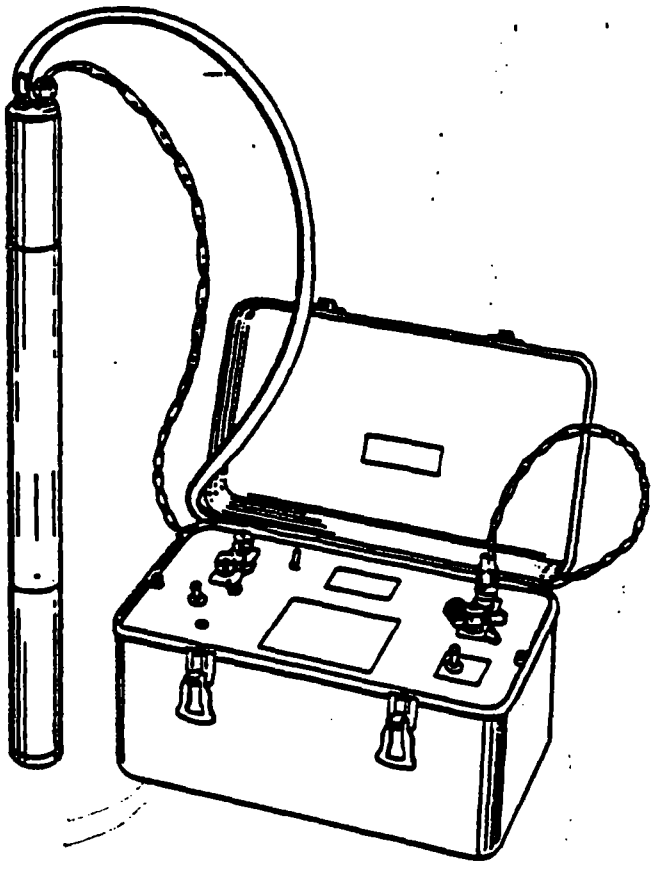


Figure 9. Johnson-Keck 2" Submersible Pump

Advantages:

- -high pumping rates allow rapid evacuation and facilitate sampling
- -can be used effectively at any depth applicable in Minnesota
- -do not need to be primed
- -permanently installed units eliminate risks of cross-contamination in sampling

Disadvantages:

- -"portable" units require use of a truck or four-wheel drive vehicle to transport the pump and generator to the sampling site (not true for 2" submersibles)
- -agitation of the sample may lead to the loss of volatile components (not true for pumps using a non-air-contact lift system)
- -commonly available pumps require at least four-inch diameter wells; pumps for two-inch wells available from only a few suppliers at present
- -sample contact with pumping mechanism may affect water quality

Usage Recommendations:

- -care should be taken when using portable systems to ensure that all parts that contact the sample are cleaned before sampling each well
- -the pump should be set above the well screen for evacuation and sampling
- -permanent installation should not be made in areas where water contaminants will corrode the pump

Suction Lift Pumps:

Suction lift pumps are appropriately used where the water to be sampled is less than 25 feet deep and volatile components are not a concern. Types of suction-lift pumps include powered perstaltic and centrifugal pumps as well as hand-operated diaphragm and pitcher pumps.

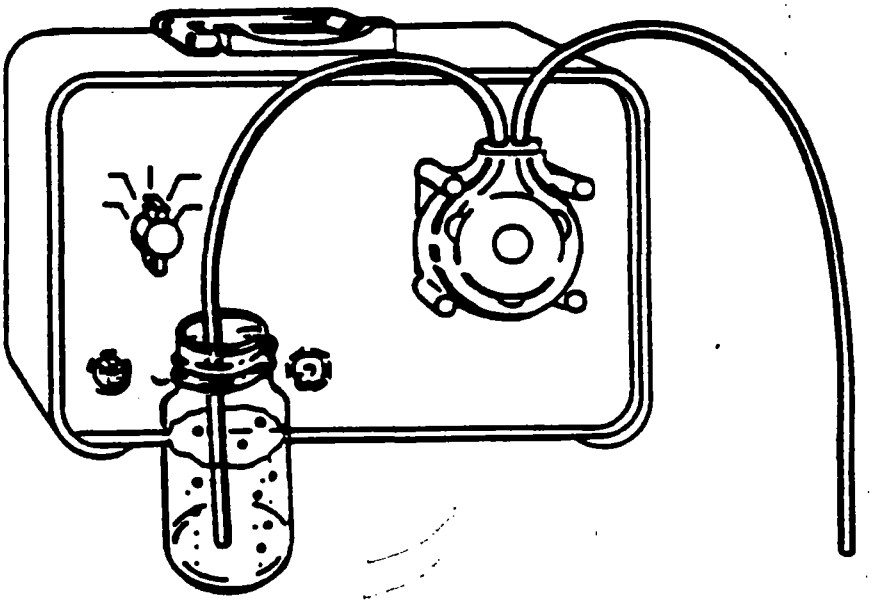


Figure 10. Masterflex Peristaltic Pump

Advantages:

- -many types made for field use
- -portable
- -relatively inexpensive
- -expedites sampling of small-diameter wells
- -fairly high pumping rates (all but peristaltics)
- -sample contacts only tubing, not pumping mechanism (peristaltic only)

Disadvantages:

- -cannot be used below the suction limit (20-30 feet of head)
- -suction and agitation may cause loss of dissolved gases
- -sample contacts pumping mechanism, which may cause changes in the parameters of interest (all types but peristaltics)

Usage Recommendations:

- -pump suction line should be set above the well screen for evacuation and sampling
- -peristaltic pumps should not be used when low (ppb) levels are to be determined
- -do not use where loss of gases and volatile components will affect the parameters of interest
- -new, cleaned tubing should be used for each sample

Air-Lift and Nitrogen-Lift Samplers:

These samplers operate by applying air pressure to a well or a sampling chamber to force a water sample out the discharge tube. This sampling technique is capable of withdrawing water from depths of at least 190 feet (Trescott and Pinder, 1970).

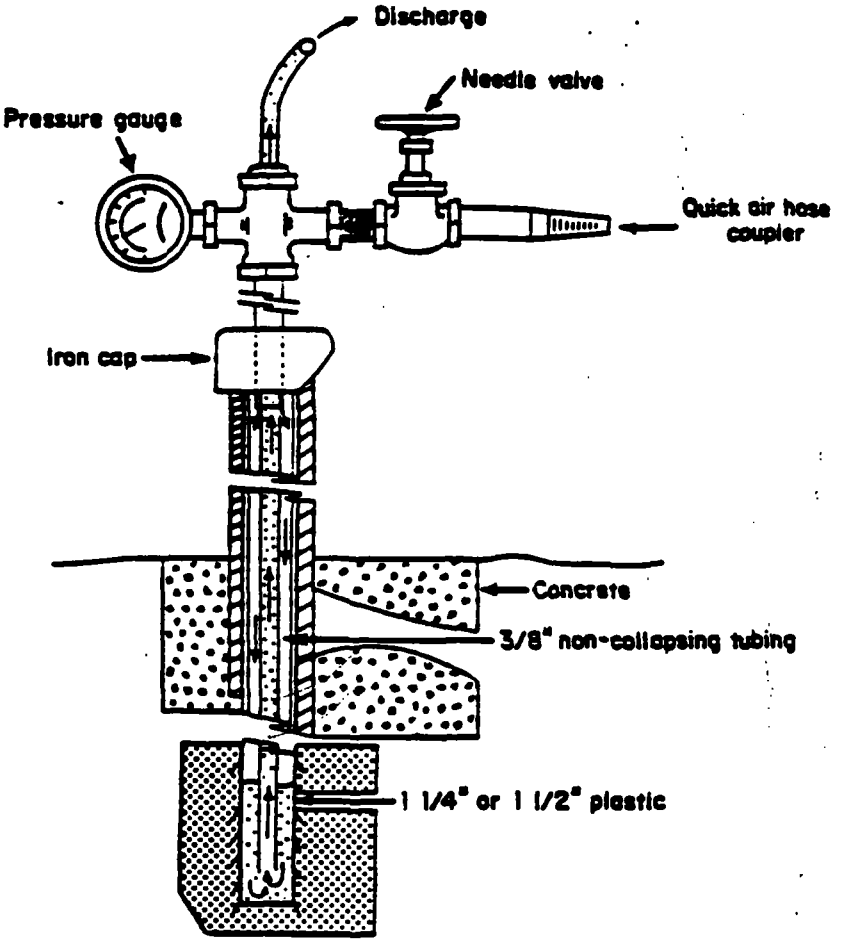


Figure 11. Air-Lift Sampler

Advantages:

-can be permanently installed, or used as a portable sampling device
-can be manufactured using easily obtainable parts

Disadvantages:

-repressurization releases dissolved CO_2 from solution, raising the pH, precipitating certain components and volatilizing others

Usage Recommendations:

-due to water chemistry changes caused using this sampling method, it is not generally recommended; those devices designed to prevent the air or nitrogen from escaping into the water in the well may be used for evacuation, with samples being taken afterward with a bailer
-when used as a portable device, care should be taken that all parts are clean prior to insertion into the well

SECTION 3

UNSATURATED ZONE MONITORING

Monitoring in the unsaturated zone may be pursued by either detection of seepage or by determination of the quality of the water seeping through the soil. Seepage detection may be of value only at sites designed for total containment systems, where any seepage constitutes a violation of permit conditions. Tensiometers, electrical resistance blocks, neutron logging or psychrometers may be used to detect seepage, triggering corrective actions aimed at eliminating any discharge from the site.

At facilities such as wastewater spray irrigation sites, the vegetation and soil are the treatment system and unsaturated flow comprising infiltration and percolation is part of the process. In these cases, samples of the soil moisture are collected and analyzed to provide information on the quality of the effluent reaching a particular depth in the soil profile.

Suction-cup lysimeters (Figure 12) are most commonly used to collect soil moisture samples although other types of lysimeters are also available. These sampling devices can be used in conjunction with monitoring wells to better define contaminant movement. Samples of the soil itself can also be analyzed for contaminants, although this is a more expensive procedure. In addition, geophysical methods such as electrical resistivity or electro-magnetic surveys may provide more information on changes in the unsaturated zone.

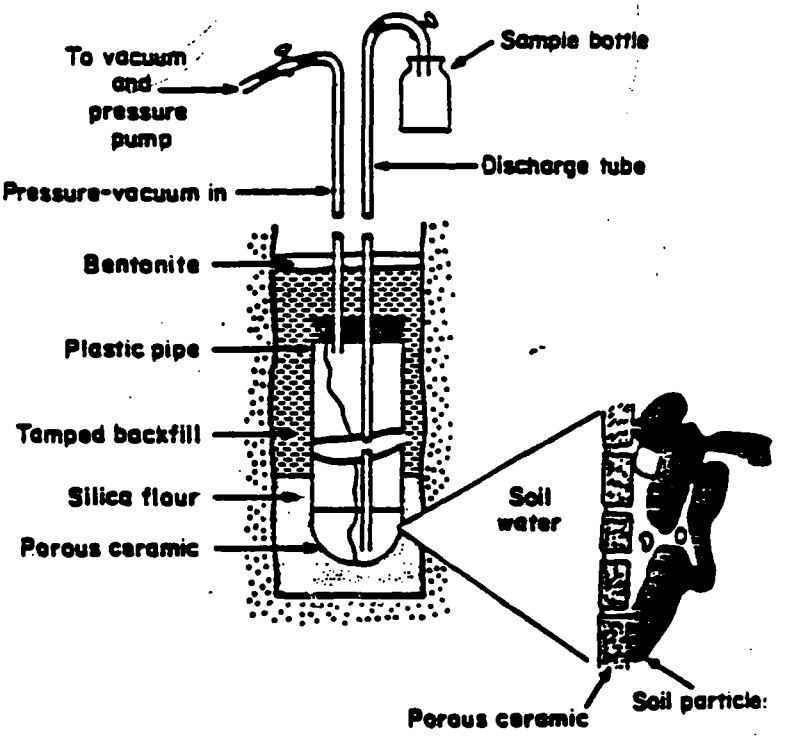


Figure 12. Suction-Cup Lysimeter

Locations for Lysimeters

Lysimeters should be placed in banks of two at each location to provide a larger volume of sample, to provide a backup system in case of failure of one of the units, and to provide a more representative sample. Both lysimeters in the bank should be installed in the same manner at the same depth. They should be placed approximately 2 feet apart.

One bank of lysimeters should be installed away from the potential contaminant source to provide background information. The others should be placed directly under the area to be monitored. For bottom-sealed sites, lysimeters should be located in the soil beneath the seal. For land disposal operations, the lysimeters should be placed in the soil under the waste area. Lysimeters should be placed below the crop root zone in spray fields.

Installation of Lysimeters

When installing suction-cup lysimeters it is important that certain details be given strict attention. These include:

- Porous ceramic samplers should be flushed with dilute acid and deionized water before use to remove contaminants left in the cup during manufacture. A vacuum should be applied to the assembled lysimeter and 250 to 500 ml. of 0.1N hydrochloric acid, followed by 500 ml. of deionized water, pulled through the ceramic wall and discarded. All parts should be completely rinsed with deionized water.

- Sampling tubes must be clearly marked with the lysimeter number as well as their purpose (sampling or vacuum) so that once they are installed, no errors will be made as to their designation.

- Assembled lysimeters should be soaked in deionized water immediately before installation so that all the pores in the ceramic cup are filled with water.

- The installation bore hole should be at least 2 inches wider than the lysimeter.

- It is essential that good contact be provided between the ceramic cup and the surrounding soil. This is best ensured by pouring enough fine silica flour slurry into the borehole to completely surround the cup, then pressing the cup firmly into the slurry.

- The bore hole should be slowly refilled with continuous tamping to eliminate any large voids. A plug of bentonite will aid in sealing off the lysimeter from direct surface infiltration.

- Surface soil should be mounded around the exposed sampling tubes, and a protective cover (such as a lockable metal box) placed over them.

- In most applications, lysimeters should not be placed within one foot of the seasonal high water table.

Sample Collection From Lysimeters

When sampling from lysimeters it is important not to apply high vacuum levels to the sampler. Some recommendations for sampling include:

-Vacuum application should be sufficient to collect enough sample for analysis. This will have to be determined on a trial-and-error basis, depending on moisture conditions, soil type, etc. A recommended starting period is one day in coarse textured soils and two days in medium to fine soils.

-Vacuum should be the lowest value that will allow sample collection, generally between 0.3 and 0.8 atmospheres (4.4 to 11.8 pounds per square inch); 0.8 atmospheric pressure usually works best for lysimeters installed with silica flour around the ceramic cup.

-The applied air pressure for sample expulsion should also be as low as possible, to avoid pushing most of the sample back out of the cup into the soil.

Lysimeter Maintenance

To facilitate collection of representative samples and maintenance of these devices, all lysimeters should have vacuums placed on them at a minimum of once a month with samples either wasted or analyzed according to monitoring requirements. Sample tubes should be clearly labeled at all times to eliminate confusion. Lysimeters should not be left under vacuum in extremely cold weather to prevent damage from freezing.

SECTION 4

SURFACE WATER MONITORING

Identifying Surface Water Monitoring Points

Surface water is monitored in instances where contaminated ground water may potentially discharge into surface water. If the surface water affected is a river or stream, monitoring should take place upstream and downstream from the waste facility and at the point of discharge. Standing bodies of water should be monitored at the apparent point of discharge and at apparently unaffected points as well. A stake or permanent marker should be placed on the bank to mark routine monitoring locations. Care should be exercised in selecting sampling points so that all possible impacts on the stream or lake are noted. For example, downstream samples for a landfill should not extend past an outfall for another discharger or ambiguous data may be generated.

A surface water monitoring plan should also be submitted to the Agency for approval prior to initiating sampling. The Agency will review the plan for completeness and appropriateness of parameters and sampling frequency.

Sampling Surface Waters

When samples are collected from a river or stream, water quality may vary with depth, stream flow, distance from shore and from one shore to the other. It is best to take an integrated sample from top to bottom in the middle of the stream. VanDorn or Kemmerer samplers can be used for this purpose. Instructions on their use are included in Appendix B.

When sampling shallow water (less than 3 feet deep), it is best to take a grab sample in the middle of the stream at mid-depth, holding the sample container under the surface until filled. The mouth of the container should face into the flow.

Sampling extremely shallow water, such as leachate seeps, can be very difficult. Care must be taken not to disturb the bottom sediments when sampling. If the site is a routine monitoring point, a small depression should be made to allow more water to collect for sampling. A carefully-held peristaltic pump sampling tube can then be used to collect the sample.

In shallow streams the sampling should start at the furthest downstream point and move upstream so that any disturbances caused by sampling do not affect the quality of the water sampled. In deeper waters such as rivers, sampling is usually done from boats or bridges, so the disturbance is minimal. For such cases, sampling should begin first at the upstream point, next to the downstream point, and finally to the sample point closest to the apparent source of discharge. In that way, the chance of contaminants clinging to the sampler is reduced.

SECTION 5

SAMPLING FROM WATER SUPPLY WELLS

When samples are required to be drawn from wells used as drinking or industrial water supplies, the following procedure should be followed:

-Samples should be taken from the point closest to the well, before the water is softened, filtered or heated.

-If possible, samples should be taken before the water enters the pressure tank. If that is not possible, let the water run to waste long enough to empty the tank and the water in storage in the pipes.

-Obtain well logs, or where not available, find out from the well owner construction details of the well, including completed depth, static water level and well diameter. This will allow the amount of water in storage in the casing to be determined. If the well is in current use, evacuation of one well volume (and the pressure tank) should be sufficient to obtain a sample of fresh aquifer water. If the well has not been used recently, a stabilization test should be performed to determine the amount of pumping necessary.

-Remove aerators, filters, or other devices from the tap before sampling.

-In some cases, it is desirable to obtain a sample of the water which represents the quality of the water which is being consumed. For this, the tap sampled should be the one most frequently used for drinking and cooking. Only is this instance is it proper to leave aerators, filters, etc. on the tap when the sample is being drawn.

SECTION 6

SAMPLING FOR ORGANICS

When sampling for volatile organics, samples should be obtained with a Teflon bailer. Wells must be properly evacuated (see stabilization tests, page 6) before sampling. Other methods of water removal may be used for the initial evacuation. Stainless steel bailers are less desirable because some organics may be absorbed by metal (Pettyjohn, 1981). The sample vials should be overfilled without entrapped air bubbles to achieve a positive meniscus, shown in Figure 13. Bottom-emptying devices on bailers will aid in sample transfer without aeration. Teflon bailers are also appropriate for obtaining grab samples for other organic compounds such as pesticides, PCB's, etc.

It is important that the well be properly evacuated prior to sampling, although any of the pumps described earlier (hexane and distilled water rinsed) can be used for that purpose. Where adequate analytical sensitivity and sample uniformity cannot be obtained by grab sampling, continuous sampling procedures are used to concentrate and recover non-volatile organic constituents from relatively large volumes of ground water. Descriptions of these procedures are available (Pettyjohn, 1981; Scalf, 1981).

Figure 13. Filled Vial for Volatile Organics

SECTION 7

FIELD MEASUREMENTS AND SAMPLE PREPARATION

Prior to evacuating the well, depth to ground water should be measured to the nearest hundredth of a foot (0.01 foot). Depth to water must be reported relative to the top of the well casing as well as the calculated water surface elevation. Weighted steel tapes are preferred for depth measurement, but other methods may be used if the desired accuracy can be obtained.

A stabilization test should be performed for each monitoring well to determine the amount of pumping required to obtain a sample of water from the formation. Section 2 of this manual discusses the stabilization test procedure in greater detail. Each well should be pumped at the rate used in the stabilization test for the period of time which was required to obtain stabilized readings. Once the well has been thus evacuated, a sample should be drawn for field analyses of pH, temperature, and specific conductance. Manufacturers' directions for usage of the pH and conductance meters should be followed. Calibration of the meters should be performed daily.

Another sample should be taken for laboratory analysis. Sample containers should be supplied by the laboratory. The container should indicate the types of analyses to be performed. Field filtration through a 0.45 micron filter should be done on that portion of the samples to be analyzed for dissolved metals. A new filter should be used for each sample, and the filtration apparatus should be cleaned between sample locations. Appropriate preservation techniques must be followed immediately upon sample collection (and after filtration for dissolved parameters) to minimize chemical changes which could affect analytical results. Appendix C shows the appropriate container types and preservation methods for various parameters.

The same field analysis, filtration, and preservation techniques should be observed for all types of samples, except that samples taken from drinking and industrial water supply wells should not be filtered. By not filtering, the analysis will show the concentration of metals in the water as it is being consumed.

All samples should be iced or refrigerated and transported to the laboratory within 24 hours of sampling. To assure prompt analysis, it is advisable to sample early in the week so that samples do not remain in the laboratory over the weekend.

SECTION 8

ESTABLISHING A SAMPLING PROTOCOL

A sampling protocol for each site should be established, maintained by the site owner/permittee, and made available to field personnel before each anticipated sampling date so adequate preparation can be made. The protocol should include the following:

-the order in which the wells and other monitoring points are to be sampled (it is generally preferable to begin with the wells containing the best quality water and end with those with the worst to lessen the chance of cross-contamination);

-methods to be followed at each sampling point, including the stabilization procedures;

-equipment to be used for water level measurement, evacuation, and sampling;

-procedures to be followed for cleaning the equipment between samples;

-the type of containers to be filled and the volume of sample needed for each;

-field testing, filtration, and preservation methods;

-the cleaning procedure and volume and type of cleaning agent to be used for cleaning the equipment between sampling points; and

-sample shipping procedures or procedures to be followed when samples are delivered to the laboratory, as appropriate.

A copy of the protocol should be kept in a field notebook. The notebook should also include accurate records of measurements, notes and comments made at each sampling event. In the Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities (USEPA, 1977), USEPA recommends that the following records be kept for each sample submitted to the laboratory:

-sample description--type (ground water, surface water), volume;

-sample source--well number, location;

-sampler's identity--chain of evidence should be maintained; each time transfer of a sample occurs, a record including signatures of parties involved in transfer should be made;

-time and date of sampling;

-significant weather conditions;

-sample laboratory number;

-pertinent well data--depth of well, depth to water table, water surface elevation, and schedule and method of pumping;

-sampling method--vacuum, bailer, pressure;

-type and number of preservatives, if any (e.g., NaOH for cyanide, H_3PO_4 and $CuSO_4$ for phenols, etc.), or whether the sample was filtered;

-sample containers--type, size, and number (e.g., three liter glass stoppered bottles, one gallon screw-cap bottle, etc.);

-reason for sampling--initial sampling of new landfill, annual sampling, quarterly sampling, special problem sampling in conjunction with contaminant discovered in nearby domestic well, etc.;

-appearance of sample--color, turbidity, sediment, oil on surface, etc.;

-any other information which appears to be significant--(e.g., sampled in conjunction with state, county, local regulatory authorities; samples for specific conductance value only; sampled for key indicator analysis; sampled for extended analysis; resampled following engineering corrective action, etc.);

-name and location of laboratory performing analysis;

-water temperature upon sampling;

-thermal preservation--(e.g., transportation in ice chest);

-analytical determinations (if any) performed in the field at the time of sampling and results obtained--(e.g., pH, temperature, dissolved oxygen, and specific conductance, etc.);

-analyst's identity and affiliation.

SECTION 9

LABORATORY REQUIREMENTS

All analyses must be performed according to USEPA approved methods listed in Methods for Chemical Analysis of Water and Wastes, or The Determination of Halogenated Chemicals in Water by the Purge and Trap Method (USEPA Method 502.1) and The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method (USEPA Method 503.1), or equivalent methods approved by USEPA. Each laboratory doing Agency-required analyses will be asked to provide the Agency with a list of methods referenced to the above documents, for all required tests.

Samples should ideally be collected by laboratory personnel. In all cases, sample containers must be provided by the laboratory. Due to the sensitivity of analyses for volatile organic compounds, it is recommended that these samples in particular be collected by experienced field or laboratory personnel only. The laboratory must have a quality assurance procedure for sample containers. The containers should be of the types specified in Appendix C. Preservatives and instructions for their use should also be provided.

The Agency is now requiring that a quality assurance plan for each site be approved before sampling begins. These plans must not only be submitted for new sites, but also for each change of laboratory at existing sites. Plans must be kept current, with the Agency notified of any change or variation in procedure. A quality assurance program is now being developed for many MPCA programs. Contact Orbbie Webber (MPCA, Solid and Hazardous Waste Division) for more information.

The following list of items must be included in a quality assurance plan:

-the order in which the wells are to be sampled and the rationale for this order;

-the amount of water in gallons and well volumes which must be evacuated from each well prior to sampling based on stabilization tests or other methods; if other methods are used, they should be detailed;

-a list of the parameters to be analyzed for in each well, a sampling schedule, and a parameter protocol;

-the methods followed and equipment used for measuring the static water level, stabilizing the well, evacuating each well, obtaining a sample, and field filtering;

-the cleaning procedures, and materials and volume of cleaning agent used for cleaning equipment between wells;

-sample preservation methods, minimum sample volumes and container specifications for each parameter;

-a sample chain-of-custody form which will be used;

-shipping and handling procedures and a time schedule from the field to the laboratory to actual analysis for each parameter;

-the methods to be used for analysis, referenced to EPA or Standard Methods;

-the lowest detection limits and reporting limits which are achieved according to the method and equipment used;

-the type of equipment used in the laboratory and its maintenance schedule;

-the number of quality control samples (blanks, spikes, or duplicates) that are proposed to be analyzed as a part of each sample set for the parameters to be analyzed in the investigation; at least one field blank must be analyzed and reported for each sample set for volatile organics analyses; and

-the scheduled first sampling date so that Agency staff has an opportunity to split samples, inspect the wells, and observe the sampling procedure with the person assigned to collect the samples.

Field checks should be performed for purposes of quality assurance. In USEPA's Handbook for Analytical Quality Control in Water and Wastewater Laboratories (March, 1979), the following checks are recommended:

-Duplicate Samples - At selected stations on a random time frame duplicate samples are collected from two sets of field equipment, or duplicate grab samples are collected. This provides a check of sampling method and equipment for precision.

-Split Samples - A representative subsample from the collected sample is removed and both are analyzed for the pollutants of interest. The samples may be reanalyzed by the same laboratory or analyzed by two different laboratories for a check of the analytical procedures.

-Spiked Samples - Known amounts of a particular constituent are added to an actual sample or to blanks of deionized water at concentrations at which the accuracy of the test method is satisfactory. The amount added should be coordinated with the laboratory, and the person performing the spike must be proficient in qualitatively performing the operation. This method provides a proficiency check for accuracy of the analytical procedures.

-Sample Preservation Blanks - Acids and chemical preservatives can become contaminated after a period of field use. The sampler should add the same quantity of preservative to some distilled water as normally would be added to a water sample. This preservative blank is sent to the laboratory for analysis of the same parameters that are measured in the sample and values for the blank are then reported along with the sample values. Chemical preservatives should be changed every two weeks, or sooner, if contamination increases above predetermined levels.

-Field Blanks - Field blanks may be necessary where sampling equipment or other factors could affect the parameters of interest. Field blanks consist of deionized water, run through clean sampling equipment, then preserved and handled in the same manner as the samples.

"Chain of custody" tracking is recommended for all samples. The following discussion of this procedure is from the Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities (USEPA, 1977).

Proper chain of custody procedures play a crucial role in enforcement cases. The following are some basic guidelines which have legal significance:

-As few people as possible should handle the sample.

-Stream and ground water samples should be obtained by using standard field sampling techniques as discussed in this manual.

-The chain of custody records should be attached to the sample container at the time the sample is collected, and should contain the following information: sample number, date and time taken, source of the sample (include type of sample and name of firm), the preservative and analysis required, name of person taking sample, and the name of any witness. The prefilled side of the card should be signed, timed, and dated by the sampler. The sample container should be sealed, with an indication of the regulatory agency's designation, date, and sampler's signature attached. The seal should cover the string or wire tie of the chain of custody record, so that the record or tag cannot be removed or the container opened without breaking the seal. The tags and seals should be filled out in legible handwriting. When transferring the possession of samples, the transferee should sign and record the date and time on the chain of custody record. Custody transfers, if made to a sample custodian in the field, should be recorded for each individual sample. The number of custodians in the chain of possession should be as few as possible. If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples should be locked in a designated area within the laboratory so that no one can tamper with them.

-Blank samples should be collected in containers with or without preservatives, so that the laboratory analysis can be performed to show that there was no container contamination.

-A field book or log should be used to record field measurements and other pertinent information necessary to refresh the sampler's memory if he later becomes a witness in an enforcement proceeding.

-A separate field notebook should be maintained for each facility. It must be stored in a safe place where it can be protected and accounted for at all times. A standard format should be established to minimize field entries. The entries should then be signed by the field sampler. The responsibility for preparing and retaining field notebooks during and after a sampling survey should be assigned to a survey coordinator or his designated representative.

-The sampler is responsible for the care and custody of the samples collected. He must assure that each container is in his possession or in his view at all times or stored in a locked place where no one can tamper with it.

-Photographs can be taken to establish exactly where the samples were obtained. Written documentation on the back of the photograph should include the signature of the photographer, the time, date, and site location. Photographs should be handled according to the established chain of custody procedures.

-Each laboratory should have a sample custodian to maintain a permanent log book in which he records for each sample: the person delivering the sample, the person receiving the sample, date and time received, source of sample, sample number, how the sample was transmitted to the lab, and a number assigned to each sample by the laboratory. A standardized format should be established for log-book entries. The custodian should insure that heat-sensitive or light-sensitive samples or other sample materials having unusual physical characteristics that may require special handling are properly stored and maintained. Distribution of samples to laboratory personnel who are to perform analyses should be made only by the custodian. The custodian should log the laboratory sample number, time, date, and the signature of the person to whom the samples were given. Laboratory personnel should examine the seal on the container prior to opening and should be prepared to testify that their examination of the container indicated that it had not been tampered with.

Figure 14 shows a chain-of-custody form currently in use by the Agency. The form is a multi-part carbonless form, with copies distributed to the sampler, laboratory and regulatory agency.

Minnesota Pollution Control Agency
Solid Hazardous Waste Division
1935 West County Road B2
Roseville, Minnesota 55113

Figure 14. Chain of Custody Form

S 05006

CHAIN OF CUSTODY RECORD

Project Name									Name of Sampler	
Field Number	Date	Time	Sample Type(s): monitoring well	existing well	surface water	wastewater	waste	other	Sample Location	Analyses Requested	Comments on Samples

Remarks on Site

Samples Relinquished by	Samples Received by	Comments	Date/Time
Samples Relinquished by	Samples Received by	Comments	Date/Time
Samples Relinquished by	Samples Received by	Comments	Date/Time
Means of Delivery		Seals intact: ☐ YES ☐ NO ☐ N.A.	

Original and yellow copies to lab. Pink copy to MPCA. Lab forwards completed yellow copy to MPCA with analytical results.

PQ-00343-01 (12/83)

SECTION 10

REPORTING REQUIREMENTS

The results of the required analyses are to be submitted to the Agency in the time frame specified in the permit, order, or agreement. Submittal of reports should be on forms provided by the Agency when so directed. Information to be reported includes brief descriptions of the following:

- -the static water level for each well to the nearest 0.01 foot from the surveyed reference point;
- -the volume of water removed before sampling;
- -sampler's field comments on unusual or noteworthy occurrences during the sampling event;
- -a statement explaining the reasons for and ramifications of any deviations in sampling or analysis techniques or equipment used from that stated in the approved quality control plan; and
- -the laboratory results of each sample analysis along with quality control sample analyses (i.e., blanks, spikes, duplicates). An analysis of a field blank for each sampling event for volatile organics must be included.

Annual reports may also be required. These should cover one calendar year and be submitted by the end of the following January. In the annual report, the facility owners or operators provide the Agency with a detailed interpretation of the monitoring results collected from each site. These reports are to be prepared by persons knowledgeable in the field of ground water pollution. In many cases, this will require the disposal site permittee to retain the services of a qualified consultant to prepare the report. Contents of the report should include:

- -a narrative, describing the effects which the site is exerting on surrounding ground water quality and any changes made or maintenance needed in the monitoring network;
- -data summary tables;
- -local rainfall conditions at the closest measurement station (information obtainable from the National Climate Data Center, Federal Building, Asheville, North Carolina 28801; 704-259-0682);
- -well hydrographs for water elevations at all monitoring points; and
- -graphics showing concentration versus time for all measured parameters, by well, for as long as the record exists. It is unnecessary to graph parameters consisting of only one data point per year or those which are consistently reported as below detectable limits.

APPENDIX A:

SAMPLING FREQUENCY; AND
SITE-TYPE GUIDELINES FOR MONITORING:

1. Community drainfields
2. Hazardous waste facilities
3. Land application sites
4. Landfills:
    a. Mixed municipal waste (sanitary) landfills
    b. Industrial waste landfills
    c. Demolition waste landfills
5. Rapid infiltration basins
6. Spill sites
7. Surface impoundments

Monitoring systems should be individually designed, taking into account a site's hydrogeologic setting. Each site will have monitoring requirements established by permit or by letter of permit amendment, giving monitoring point locations, sampling frequency, and required parameters. Monitoring systems should be designed to provide the information necessary to effectively evaluate any impacts which the facility may exert upon the ground water. For more information on the design of monitoring systems to meet Agency requirements, see "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985).

The remainder of this appendix lists general guidelines for ground water monitoring at various types of waste management facilities in terms of required parameters and sampling frequency. Ideally, sampling should be timed to coincide with the spring thaw, the summer peak of evapotranspiration, and the autumn dry season prior to freeze-up. This manual takes a new approach, by dividing the State into sampling regions and specifying a seasonal monitoring schedule for each region. The regions are shown in Figure A-1, and the schedule given in Table A-1. Facilities with year-round hydraulic loading, such as community drainfields, will still be required to adhere to a quarterly monitoring schedule.

Table A-2, which appears at the end of this appendix, provides a summary of monitoring parameters for various waste management facility types.

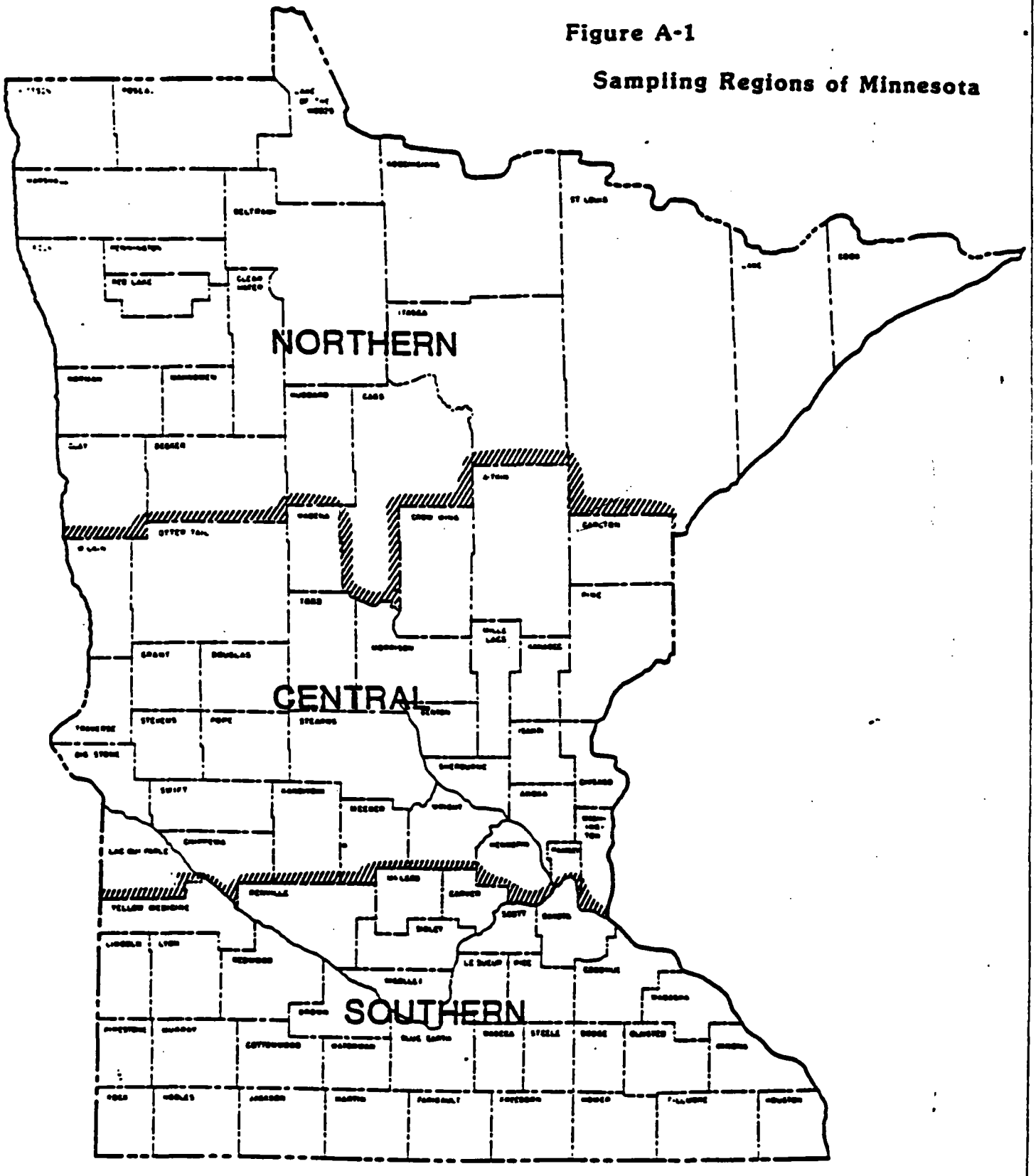
Table A-1

Monitoring Schedule for Seasonal Frequency

	Spring	Summer	Autumn
Southern 1/3	March 14-April 21	June 21-July 31	October 21-November 21
Central 1/3	March 28-April 28	July 1-August 7	October 14-November 14
Northern 1/3	March 28-May 14	July 1-July 31	October 1-October 31
Submit Reports By	July 31	October 31	With Annual Report by January 31

Figure A-1

Sampling Regions of Minnesota



COMMUNITY DRAINFIELDS

Large drainfields designed for loading rates greater than or equal to 5000 gallons per day must monitor ground water to meet Agency requirements. Those designed for loading rates less than 5000 gallons per day and greater than 1200 gallons per day will be required to monitor if a case-by-case review of the system shows it to be necessary. Also, if a land development project proposed on-site systems which individually were less than 1200 gallons per day but with a cumulative loading over a given area which would be equivalent to a larger system, then the combined effects of that project would also need to be evaluated.

Monitoring systems should be designed to provide the information necessary to effectively evaluate any impacts which the system may exert upon the ground water. For more information on the design of monitoring systems to meet Agency requirements, see "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985). A detailed guidance manual on community drainfields has also been prepared by the Agency, entitled "High Rate Soil Absorption System Task Force Final Report" (MPCA, 1984).

Samples should be collected in the months of March, June, and September by the methods described in this manual. Parameters to be analyzed include:

Alkalinity
Chemical Oxygen Demand
Chloride
Fecal Coliforms
Hardness
Influent Flow
Inspection Pipe Observation
Nitrogen, Ammonia
Nitrogen, Kjeldahl
Nitrogen, Nitrate
pH(1)
Specific Area of Application
Specific Conductance(2)
Sulfate
Temperature(2)
Volatile Organics
Water Elevation(3)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) As measured in field before pumping or bailing.

Analyses should be done in accordance with the requirements of this manual, pages 26-30.

In the event that the system is no longer used, monitoring should continue on a quarterly basis for at least one year, when the Agency will evaluate the need for continued monitoring. If no serious impacts are detected, monitoring may cease at that time. All wells should be abandoned in accordance with the Minnesota Water Well Construction Code when monitoring ceases.

HAZARDOUS WASTE FACILITIES

Environmental monitoring is critical at hazardous waste facilities. For that reason, the rules governing hazardous waste management are very specific regarding monitoring. Federal requirements are presented in 40 CFR Parts 264 and 265 (Federal Register, Volume 47, Number 143, July 26, 1982) and State of Minnesota requirements in Minnesota Rules Chapter 7045 distributed by the Department of Administration, Documents Section, 117 University Avenue, St. Paul, Minnesota 55155.

Ground water monitoring design, construction and implementation at uncontrolled hazardous waste sites is subject to review by the Agency. Procedures for monitoring uncontrolled sites are determined on a case-by-case basis. Contact the Agency for additional information.

LAND APPLICATION SITES

Minnesota Rule Chapters 7040.0100-7040.4700 regulates the landspreading of municipal sewage sludge. If sewage sludge is applied to land which is owned, leased, or rented by the political subdivision generating the sludge (classified a landspreading facility), ground water monitoring may be required. The rules specify the background parameters to be analyzed for and the depth and location of monitoring wells. Sampling frequency and analytical parameters will be determined on a case-by-case basis.

Monitoring at land application facilities for effluents is determined by the hydraulic loading rate. In general, systems where hydraulic loading rates are based on either crop irrigation requirements (consumptive use) or nitrogen requirements will not be required to monitor ground water. Exceptions to this generality would be systems located on extremely coarse soils, over shallow ground water, or close to downgradient drinking water wells, or if the wastewater contains a parameter at a concentration which may impact ground water use.

Land application facilities which apply wastewater in excess of crop irrigation and nitrogen requirements will be required to monitor ground water. Monitoring systems should be designed to provide the information necessary to effectively evaluate any impacts which the system may exert upon the ground water. For more information on the design of monitoring systems to meet Agency requirements, see "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985). Minimum monitoring parameters include:

Ammonia Nitrogen
Chloride
Nitrate + Nitrate Nitrogen, as N
pH(1)
Specific Area of Application
Specific Conductance(2)
Water Elevation(3)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) As measured in field before pumping or bailing.

If the water table elevations are very deep at a land application site or if it is necessary to detect downward movement of specific wastewater components before they reach the water table, lysimeter installation may be required. The placement of lysimeters will be dependent on soil characteristics, crop rooting depth, water table elevation and behavior of parameters of concern in the soil environment.

At a minimum, sampling frequency for monitoring wells will be as described for seasonal monitoring described earlier in this appendix. More frequent sampling may be required based on hydraulic loading rates, length of spray

season, soil permeability, ground-water flow direction, ground-water flow velocity and concentration of specific parameters of concern in the wastewater.

Post-operative monitoring should continue seasonally for two years after spraying is discontinued. At this time the Agency will reevaluate the monitoring program and make recommendations for either increasing or decreasing monitoring requirements. Following the initial post-closure reevaluation, monitoring should continue as directed by the Agency, or until one year of monitoring data shows no contamination (see page 5), whereupon monitoring may cease at the site. All wells should be abandoned in accordance with the Minnesota Water Well Construction Code when monitoring ceases.

MIXED MUNICIPAL WASTE (SANITARY) LANDFILLS (INCLUDING MODIFIED SANITARY LANDFILLS

All sanitary landfills in Minnesota are required to monitor ground water. Their monitoring systems should be designed to provide the information necessary to effectively evaluate any impacts which the facility may exert upon the ground water. For more information on the design of monitoring systems to meet Agency requirements, see "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985).

New wells, including both those at new and existing facilities, should be sampled for the following parameters to establish the initial quality of the ground water.

Alkalinity	Manganese, Dissolved
Ammonia Nitrogen	Mercury, Dissolved
Arsenic, Dissolved	Nitrate + Nitrite, as N
Cadmium, Dissolved	pH(1)
Calcium, Dissolved	Potassium, Dissolved
Chemical Oxygen Demand	Sodium, Dissolved
Chloride	Specific Conductance(2)
Chromium, Total Dissolved	Sulfate
Copper, Dissolved	Suspended Solids, Total
Dissolved Solids, Total	Temperature(2)
Iron, Dissolved	Volatile Organics(4)
Lead, Dissolved	Zinc, Dissolved
Magnesium, Dissolved	Water Elevation(3)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) As measured in field before pumping or bailing.
(4) Halogenated and non-halogenated, purge-and-trap method.

One sampling event is not sufficient to establish background water quality. Seasonal variation, sampling variation, analytical variation and random error occur. Monitoring wells at new sites will be required to have three background samples. The first sample will require analysis of the extended list of parameters for each well. Within 30 days after this sample is taken the analytical results should be submitted to the Agency. At least two more background samples taken at one month intervals will be required for each well. However, based on the results from the first sampling round, the Agency may reduce the number of parameters required from the extended list or extend the intervals between sampling events. For new wells at existing or expanded sites, three samples analyzed for the extended list will be required to establish initial water quality. The sampling interval will be based on the purpose of the well within the monitoring program and will range from monthly to seasonal, coinciding with the existing monitoring program.

The extended list is to be repeated at least every other year. Certain parameters such as volatile organics may be required more frequently.

Routine sampling should occur seasonally, as described earlier in this appendix. In addition to the three routine monitoring periods yearly, an annual report is due in January which describes the monitoring results for the previous year. See Section 10 of this manual for more information on contents of the annual report.

Parameters required for routine monitoring include, at a minimum:

Ammonia Nitrogen
Chemical Oxygen Demand
Chloride
Iron, Dissolved
Nitrate + Nitrite, as N
pH(1)
Specific Conductance(2)
Sulfate
Temperature(2)
Water Elevation(3)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) As measured in field before pumping or bailing.

All samples should be obtained following methodology and analytic techniques of this manual.

Routine monitoring should continue for five years after the site is closed, whereupon the monitoring program will be reevaluated by the Agency. Recommendations will be made to either increase or decrease monitoring

requirements. Following the initial post-closure reevaluation, monitoring should continue as directed by the Agency for a period of 15 additional years (20 total years post-closure). If no contamination is detected during this period, monitoring may cease at the site. All wells should be abandoned in accordance with the Minnesota Water Well Construction Code when monitoring ceases. NOTE: Post-closure requirements may change when the rules regulating solid waste disposal are changed in 1985.

INDUSTRIAL WASTE LANDFILLS

The monitoring requirements for industrial waste landfills will be determined on a case-by-case basis by the Agency based upon such factors as:

1. Site characteristics,
2. Waste reactivity, and
3. Present and potential downgradient water uses.

DEMOLITION WASTE LANDFILLS

Monitoring will be required at demolition landfills on a case-by-case basis, depending on the waste type, site size, operational history, and controls on dumping. If required to monitor, the guidelines for sanitary landfills should be followed.

RAPID INFILTRATION BASINS

In some cases, municipal wastewater which has received at least primary treatment may be discharged to rapid infiltration basins. The nature of these systems is such that ground water impacts may be significant, and therefore monitoring is required.

Monitoring systems should be designed to provide the information necessary to effectively evaluate any impacts which the system may exert upon the ground water. For more information on the design of monitoring systems to meet Agency requirements, see "Guidelines for Ground Water Monitoring System Design" (MPCA, 1985). A sufficient number of piezometers are also necessary to assess the dimensions (height and areal extent) of the ground water mound. Water elevations should be monitored during loading events to assess the dimensions of the ground water mound and allow for operational modifications to the system, if necessary. Water quality testing may be required as frequently as monthly. Samples should be obtained following the procedures given in this manual. Parameters to be analyzed can include:

Alkalinity
Ammonia Nitrogen
Chemical Oxygen Demand
Chloride
Nitrate + Nitrite, as N
pH(1)
Specific Designation of Basins Loaded
Specific Conductance(2)
Temperature(2)
Volatile Organics(3)
Water Elevation(4)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) Halogenated and non-halogenated, purge-and-trap method.
(4) As measured in field before pumping or bailing.

Analyses should be performed in accordance with the requirements of this manual, pages 26-30.

Monitoring should continue for at least one year after site closure, when the Agency will evaluate the need for continued monitoring. All wells should be abandoned in accordance with the Minnesota Water Well Construction Code when monitoring ceases.

SPILLS

Monitoring may be required at spill sites if the material spilled is hazardous, if seepage into ground water is suspected, or if a potential exists for negative impacts on water supplies. In these instances, the need for continuing monitoring will be evaluated on a case-by-case basis, following review of site-specific hydrogeologic information.

SURFACE IMPOUNDMENTS

Currently, all agricultural, municipal, and industrial ponds which are required to have National Pollutant Discharge Elimination System or State Disposal System permits and which seep, as evidenced by relatively small or no surface discharges must perform a water balance study. Also, a water balance must be conducted on newly-constructed ponds to verify that construction criteria have been met. Instructions for conducting a water balance study are given in the Agency document "Design/Operability Handbook," currently in draft form.

A ground water impact study is required when the water balance study indicates seepage in excess of 3500 gallons per acre per day if the system was approved prior to May 16, 1975. Monitoring may also be required at sites which have less than 3500 gallons per acre per day based on pollution potential from that particular site. Facilities approved after that date will be individually reviewed to determine the appropriate action.

Monitoring wells should be installed around the perimeter of the stabilization pond system and downgradient from each pond. A sufficient number of wells should be installed to determine background water quality. In addition, the approximate area downgradient that could potentially be affected by seepage must be established.

Samples should be obtained from the wells following methods and analytical techniques of this manual.

Initial sampling should occur at least monthly for a three-month period. Routine monitoring is then conducted on a quarterly basis, if required. For municipal and agricultural impoundments, the parameters to be analyzed include:

Alkalinity
Ammonia Nitrogen
Chemical Oxygen Demand
Chloride
Fecal Coliform
Hardness
Nitrate + Nitrite, as N
pH(1)
Sodium
Specific Conductance(2)
Sulfate
Temperature(2)
Total Kjeldahl Nitrogen
Total Phosphorus
Water Elevation(3)

(1) Two measurements: in field, immediately after obtaining sample and in laboratory.
(2) As measured in field.
(3) As measured in field before pumping or bailing.

Waste-specific parameters should be included in the monitoring of industrial impoundments. The Agency will review the data and determine appropriate follow-up action. Often, this action may involve upgrading the pond seal so that it meets the standard for new ponds.

Table A-2

— Summary of Monitoring Parameters

	Solid Waste Landfill Routine Monitoring	Solid Waste Landfill Extended Monitoring	Community Drainfield	Land Application Site	Rapid Infiltration Basin	Surface Impoundment (Wastewater)
Alkalinity		X	X		X	X
Arsenic, Dissolved		X				
Cadmium, Dissolved		X				
Calcium, Dissolved		X				
Chemical Oxygen Demand	X	X	X		X	X
Chloride	X	X	X	X	X	X
Chromium, Dissolved		X				
Copper, Dissolved		X				
Fecal Coliforms			X		X	X
Hardness			X		X	X
Influent Flow			X		X	X
Inspection Pipe Observation			X			
Iron, Dissolved	X	X				
Lead, Dissolved		X				
Magnesium, Dissolved		X				
Manganese, Dissolved		X				
Mercury, Dissolved		X				
Nitrogen, Ammonia	X	X	X	X	X	X
Nitrogen, Kjeldahl			X		X	X
Nitrogen, Nitrate + Nitrite	X	X	X	X	X	X
pH	X	X	X	X	X	X
Phosphorus, Total						X
Potassium, Dissolved		X				
Sodium, Dissolved		X				X
Solids, Total Dissolved		X				
Solids, Total Suspended		X				
Specific Area of Application			X	X	X	
Specific Conductance	X	X	X	X	X	X
Sulfate	X	X	X		X	X
Temperature	X	X	X		X	X
Volatile Organics		X	X		X	
Zinc, Dissolved		X				
Water Elevations	X	X	X	X	X	X

APPENDIX B:

USE OF THE VANDORN AND KEMMERER SAMPLERS

(Excerpted from "Quality Control Manual"
an in-house Minnesota Pollution Control Agency document)

VANDORN SAMPLER

The VanDorn sampler is designed for use in the deep waters of oceans and lakes. The Minnesota Pollution Control Agency (MPCA) uses two types of VanDorn samplers: 1) the vertical model, and 2) the horizontal model. The vertical model is used for standard sampling while the horizontal model is used for sampling near the bottom and thermoclines. The VanDorn samplers used by the MPCA are all made of plastic. They are cylindrical in shape and have capacities of 2.2, 4.2 and 30.2 liters. All models have a drain on the bottom for drawing off samples.

Through the middle of the VanDorn sampler there is a flexible rubber tube which is connected to the two end stoppers. The end stoppers have a trip wire on each stopper. The trip wire hooks onto the trip mechanism. The sounding line is attached to the trip mechanism.

The VanDorn sampler should be cleaned and then wiped dry before storage. To prevent damage to the sampler a wooden case is provided for transport and storage. The rubber tube used to close the end stoppers should be replaced yearly.

To set the sampler, pull one of the end stoppers out and attach the trigger wire to the trigger mechanism. Repeat this step with second stopper. Slowly lower sampler down to the desired sampling depth. Send down the messenger to trip the trigger mechanism. Bring the sampler to the surface. Problems may be incurred during rough water conditions which may cause drifting of the boat and subsequent failure of the tripping mechanism due to non-vertical profile of sounding line.

It is advisable before actual sampling to make sure the sampler works properly. Make sure to use a flat-nosed brass messenger, 11 ounces or smaller and a 3/16" or 1/4" braided nylon line attached securely to the sampler. Use of any other messenger or line may result in damage or loss of the sampler. If possible sampler should be allowed to soak in the lake or stream to be sampled for at least 15 minutes prior to sampling and kept in the sample water or kept closed between sample stations.

KEMMERER SAMPLER

The Kemmerer sampler is a basic water or wastewater sampler. It is designed to collect samples at specific depths.

The Kemmerer is a long cylindrical tube constructed of metal or plastic with rubber stoppers at each end. The stoppers and an associated mechanical device enables the operator to lower the open sampler to a pre-determined depth, close the sampler, and retrieve a sample from that depth.

The metal samplers are usually used for collection of samples for the common physical, and chemical determinations including pesticides. The plastic models, while satisfactory for the foregoing parameters except pesticides, are usually used for collection of samples for trace metals. The Minnesota Pollution Control Agency (MPCA) has Kemmerer samplers of 1.2, 3.1, and 6.1 liter capacities.

Whenever possible, the Kemmerer should be transported in the case that is provided, and care should be taken during handling to avoid damage. The Kemmeer should be cleaned thoroughly, then wiped down with a clean cloth before storage.

The Kemmerer sampler is used as follows:

1. Pull open the top stopper (this will also lock open the bottom stopper).

2. Rinse sampler with distilled water or in the water to be sampled before lowering the device.

3. Lower the sampler to the desired depth and close the sampler by dropping the messenger down the line.

4. Retrieve the sampler and empty contents into sampler container.

Before sampling it is important to make sure the line is tied securely to the sampler, and that the other end of the line is fastened to guard against loss of the sampler. It is good practice to take a trial sample before actual sampling starts to make sure the sampler works properly. When sampling for trace metals the plastic Kemmerer should be used, and it should be washed down with de-ionized water before each use. A 3/16" or 1/4" braided nylon sound line should be used with a flat-nosed 11 ounce or smaller messenger. Use of any other lines of messengers may result in damage or loss of the equipment.

APPENDIX C:

RECOMMENDATION FOR SAMPLE CONTAINERS AND PRESERVATION OF SAMPLES

from: "Handbook for Sampling and Sample Preservation of Water and Wastewater" U.S. Environmental Protection Agency EPA-600/4-82-029, September 1982

NOTE: These recommendations have not yet been formally adopted by the U.S. Environmental Protection Agency

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Bacterial Tests			
Coliform, fecal and total	P,G	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	6 hours
Fecal streptococci	P,G	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	6 hours
Inorganic Tests			
Acidity	P,G	Cool, 4°C	14 days
Alkalinity	P,G	Cool, 4°C	14 days
Ammonia	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Biochemical oxygen demand	P,G	Cool, 4°C	48 hours
Biochemical oxygen demand, carbonaceous	P,G	Cool, 4°C	48 hours
Bromide	P,G	None required	28 days
Chemical oxygen demand	P,G	H_2SO_4 to pH < 2	28 days
Chloride	P,G	None required	28 days
Chlorine, total residual	P,G	None required	Analyze immediately
Color	P,G	Cool, 4°C	48 hours
Cyanide, total and amenable to chlorination	P,G	Cool, 4°C NaOH to pH > 12 0.6 g ascorbic acid[6]	14 days[9]
Fluoride	P	None required	28 days
Hardness	P,G	HNO_3 to pH < 2	6 months
Hydrogen ion (pH)	P,G	None required	Analyze immediately
Kjeldahl and organic Nitrogen	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Metals[4]			
Chromium VI	P,G	Cool, 4°C	24 hours
Mercury	P,G	HNO_3 to pH < 2	28 days
Metals, except above	P,G	HNO_3 to pH < 2	6 months
Nitrate	P,G	Cool, 4°C	48 hours
Nitrate-nitrite	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Nitrite	P,G	Cool, 4°C	48 hours
Oil and grease	G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Organic carbon	P,G	Cool, 4°C HCl or H_2SO_4 to pH < 2	28 days
Orthophosphate	P,G	Filter immediately Cool, 4°C	48 hours
Oxygen, Dissolved Probe	G bottle and top	None required	Analyze immediately
Winkler	G bottle and top	Fix on site and store in dark	8 hours
Phenols	G only	Cool, 4°C H_2SO_4 to pH < 2	28 days
Phosphorus (elemental)	G	Cool, 4°C	48 hours
Phosphorous, total	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 days
Residue, total	P,G	Cool, 4°C	7 days
Residue, Filterable	P,G	Cool, 4°C	7 days
Residue, Non-filterable (TSS)	P,G	Cool, 4°C	7 days
Residue, settleable	P,G	Cool, 4°C	48 hours
Residue, volatile	P,G	Cool, 4°C	7 days

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (continued)

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Silica	P	Cool, 4°C	28 days
Specific conductance	P,G	Cool, 4°C	28 days
Sulfate	P,G	Cool, 4°C	28 days
Sulfide	P,G	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9	7 days
Sulfite	P,G	Cool, 4°C	Analyze immediately
Surfactants	P,G	Cool, 4°C	48 hours
Temperature	P,G	None required	Analyze immediately
Turbidity	P,G	Cool, 4°C	48 hours
Organic Tests[5]			
Purgeable halocarbons	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	14 days
Purgeable aromatics	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6] HCl to pH < 2[10]	14 days
Acrolein and acrylonitrile	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6] Adjust pH to 4-5[11]	14 days
Phenols	G, Teflon-lined septum	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Benzidines	G, Teflon-lined septum	Cool, 4°C 0.008%$Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction

CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES (continued).

Parameter	Container[1]	Preservative[2, 12]	Maximum Holding Time[3]
Phthalate esters	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
Nitrosamines[7]	G, Teflon-lined cap	Cool, 4°C store in dark 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
PCB's	G, Teflon-lined cap	Cool, 4°C pH 5-9	7 days until extraction, 40 days after extraction
Nitroaromatics and isophorone	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days, after extraction
Polynuclear aromatic hydrocarbons	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6] store in dark	7 days until extraction, 40 days after extraction
Haloethers	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Chlorinated hydrocarbons	G, Teflon-lined cap	Cool, 4°C	7 days until extraction, 40 days after extraction
TCDD	G, Teflon-lined cap	Cool, 4°C 0.008% $Na_2S_2O_3$[6]	7 days until extraction, 40 days after extraction
Pesticides Tests			
Pesticides	G, Teflon-lined septum	Cool, 4°C pH 5-9[8]	7 days until extraction, 40 days after extraction
Radiological Tests			
Alpha, beta and radium	P,G	HNO_3 to pH < 2	6 months

Saines, M. Errors in Interpretation of Ground Water Level Data. 1981. Ground Water Monitoring Review. v.1, no. 1. pp. 56-61.

Scalf, M.R., J.F. McNabb, W.F. Dunlap, R.L. Crosby, and J. Fryberger. 1981. Manual of Ground Water Sampling Procedures. NWWA/EPA Series.

Timco Manufacturing Company, Inc., Prairie du Sac, Wisconsin. Product literature obtained from the manufacturer. August 1982.

Trescott, P.C., and G.F. Pinder. 1970. Air Pump for Small-Diameter Piezometers Ground Water. v.6, no. 2. pp. 9-11.

U.S. Department of Agriculture. 1977. Design, Installation and Use of Porous Ceramic Samplers for Monitoring Soil-Water Quality. Technical Bulletin number 1562.

U.S. Environmental Protection Agency. 1976. Monitoring Ground Water Quality: Monitoring Methodology. EPA/530/SW-611.

U.S. Environmental Protection Agency. 1977. Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. EPA/530/SW-611.

U.S. Environmental Protection Agency. 1979a. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. EPA-600/4-79-019.

U.S. Environmental Protection Agency. 1979b. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. Environmental Protection Agency. 1981a. The Determination of Halogenated Chemicals in Water by the Purge and Trap Method, Method 502.1. EPA-600/4-81-059.

U.S. Environmental Protection Agency. 1981b. The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method, method 503.1. EPA-600/4-81-057.

U.S. Environmental Protection Agency. 1982. Handbook for Sampling and Sample Preservation of Water and Wastewater. EPA-600/4-82-029.

U.S. Geological Survey. 1976. Guidelines for Collection and Field Analysis of Ground Water Samples for Selected Unstable Constituents. Stock Number 024-001-02379-4.

APPENDIX B

STANDARD OPERATING PROCEDURES

INDEX OF STANDARD OPERATING PROCEDURES

SOP NUMBER	SUBJECT	NO. OF PAGES
LM-RMA-3024	Determination of Low Level (Part Per Trillion) PAH and Heterocycles in Water	18
LM-RMA-1112	Total Recoverable Phenolics - City of St. Louis Park	16

Enseco
A Corning Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 18

DETERMINATION OF LOW LEVEL (PART PER TRILLION) PAH AND HETEROCYCLES IN WATER

SOP No.:	Revision No.:	Effective Date:
LM-RMA-3024	7.0	September 14, 1992

Supersedes: N/A

1. Summary of the Method

This method has been designed for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion level (ppt,ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 1 can be quantitatively determined using this analytical method.

Prepared by: Phil Tallarico	Date: September 14, 1992
Management Approval:	Date: 9-14-92
QA Officer Approval:	Date: 9-14-92

This method has three options for the extraction of the samples depending on the sample type. The three options include two low and one medium level extraction. The low level options have typical reporting limits of 10.0 ppt, with higher surrogate and spike levels in one of the options to accomodate dilutions. The medium level option is eighty times higher in detection limits. A volume of sample dependent of the extraction option chosen is extracted with methylene chloride. Analysis of concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

2. Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which contains the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not the specified limits, then interferences may be present.

3. Apparatus and Materials

3.1 Glassware

Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with methanol.

Glassware should then be solvent rinseds with toluene, acetone and methylene chloride, after extensive rinsing glassware should be air dryed, then sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants.

Store glassware inverted or capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.1.1 Separatory funnel - 2000 and 4000 mL, with Teflon stopcock or continuous liquid liquid extractor, 2000 mL.

3.1.2 Drying column - glass funnel with ~10 cm anhydrous sodium sulfate.

3.1.3 Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.

3.1.4 Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).

3.1.5 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs or clips.

3.1.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent) is suitable.

3.1.7 Micro reaction vessels, 2.0 mL (Supelco 3-3295).

3.2 Gas Chromatograph

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

3.3 Column

A DB-625.5 30 meter fused silica capillary column, or equivalent.

3.4 Mass Spectrometer

A mass spectrometer operating at 70 ev (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

4. Reagents

4.1 Reagent water

Reagent water is defined as water in which the target compounds are not observed at or above the method detection limit.

4.2 Solvents

Acetone, methanol, methylene chloride, cyclohexane - Burdick & Jackson, distilled in glass, or equivalent.

4.3 Sodium sulfate

(ACS) Granular, anhydrous. Purify by heating at 400^{0}C for 4 hours in a shallow tray.

4.4 Surrogate Spiking Solution

Depending on the extraction option chosen low, low75, or medium a surrogate solution is made by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone and added to the sample prior to extraction with

methylene chloride. The compounds in the surrogate solutions are naphthalene-d8, fluorene-d10, and chrysene-d12. The low surrogate solution is at 20 ng/mL and 0.5 mL per liter of sample is added. The low75 is at 150 ng/mL and 0.5 mL per liter is added. The medium surrogate solution is at 1000 ng/mL and 1.0 mL is added to the 500 mL sample.

4.5 Internal Standard Solutions

A solution containing ca. 400 ng/mL of each internal standard is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methylene chloride. Fifty microliters of this solution is added to the 0.5 mL extract prior to analysis to give a concentration of the internal standards in the extract of 40 ng/mL.

4.6 Matrix Recovery Standard Spiking Solution

A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone. The concentrations of the spiking solution for both the low and medium level extractions are shown below:

Compound	Low Spiking Solution (ng/mL)	Medium Spiking Solution (ng/mL)	Low75 Spiking Solution (ng/mL)
Naphthalene	20	1000	150
Fluorene	20	1000	150
Chrysene	20	1000	150
Indene	20	1000	150
Quinoline	20	1000	150
Benzo(e)pyrene	20	1000	150
2-methylnaphthalene	20	1000	150

The low spiking solution is added at 0.5 mL per liter of sample. The low75 is added at 0.5 mL per 1.0 liter of sample. The medium level spiking solution is added at 1.0 mL per 500 mL of sample.

5. Sample Preservation, Storage and Holding Times

5.1 Sample Preservation and Storage

The samples must be protected from light and refrigerated at 4°C (± 2°C) from the time of receipt until extraction and analysis. After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (± 2°C).

5.2 Holding Times

Samples must be extracted within 5 days of the time of sample receipt. Samples are required to be shipped the same day samples are collected using an overnight carrier.

Extracts must be analyzed within 40 days of extraction.

6. Sample Extraction

6.1 Samples

Samples are extracted at a pH>12. For the low level extraction, a measured amount of sample, approximately 4 liters, is poured into either two 2-liter continuous liquid-liquid extractors, one 4-liter continuous liquid-liquid extractor, or two 4 liter separatory funnels. The surrogate solution is added and the samples are extracted with methylene chloride. The samples are shaken three times with 80 mL of methylene chloride for the shakeout technique. The samples are allowed to reflux for eighteen hours if the liquid-liquid extractor technique is used for preparation. The extracts from each two-liter fractional extraction (for either technique) are then combined for concentration. The medium level extraction requires that 500 mL of the sample be extracted with methylene chloride for 18 hours in a one liter continuous liquid-liquid extractor or shaken three times with 60 mLs of methylene chloride in a 2-liter separatory funnel. The extracts are passed through an anhydrous sodium sulfate drying column into a 500 mL Kuderna-Danish evaporative concentrator.

Both low level extracts are concentrated to approximately 0.5 mL and transferred to a 2.0 mL microreaction vessel. The methylene chloride is evaporated using a nitrogen stream. The evaporative concentrator tube is successively rinsed with methylene chloride, the rinsates added to the reaction vessel and the methylene chloride again evaporated. This process is continued until at least five (5) 1 mL rinsings of the tube have occurred.

The final methylene chloride extract for the low level extraction is evaporated to 500 uL. All microreaction vessels are permanently marked at the 500 uL level and additional methylene chloride added, when necessary, to insure a final 500 uL extract volume. The medium level extract is concentrated to 5.0 ml using the same procedure described above. The extract vessels are capped with a Teflon fitted septum cap and stored at 4°C prior to GC/MS analysis.

6.2 Method blank

Method blanks are prepared by treating a 4-L or 500 ml of laboratory reagent water exactly as described above depending on the option chosen. A method blank must be performed once each case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix or whenever samples are extracted by the same procedure, whichever is most frequent.

* A case is a group or a set of samples collected from a particular site over a given period of time.

6.3 Matrix Recovery Sample

Matrix recovery samples are prepared by spiking a sample as described in section 4.6. The fortified sample is extracted exactly as described above for samples. The laboratory will spike and analyze 5% matrix spike samples (i.e. one matrix spike with every 20 samples).

6.4 Duplicate Sample

For a minimum of 10% of the samples analyzed a duplicate sample will be taken at sampling and a duplicate analysis will be performed. This will be carried out to insure that an estimate of precision will be available.

7. GC/MS Calibrations

Prior to use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of analysis. Only one level of calibration is used for the two low level and the medium level ppt PAH analyses. The concentrations of standards used to construct the calibration curve are 20, 40, 240, 600, and 1200 ng/mL. The linear range for low level analysis (4 L to 0.5 mL) corresponds to sample concentrations of 2.5, 5, 30, 75, and 300 ng/L. If the concentration of any target compound in a sample exceeds the linear range defined by the above standards, the extracts must be diluted so that the most concentrated analyte falls within the upper half of the calibration curve. The linear range for medium low level analysis (0.5 L to 5.0 mL) corresponds to final sample concentrations of 200, 400, 2400, 6000 and 12000 ng/L. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH relative to the internal standard is determined, as described in the Calculations section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ±35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Table 2 contains example RRT data for target compounds.

8. Daily GC/MS Performance Tests

The GC/MS will not be tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. EPA has dropped this requirement for selected ion monitoring (SIM) methods. This allows the laboratory to tune the instrument to maximize the sensitivity for the compounds being analyzed as described below.

Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. In the FC43 spectra, the ion abundance of masses 131 and 219 are adjusted to a approximate ratio of 1:1. These two ions are then maximized to be approximately 50 to 70% of the ion abundance of the base mass 69. This procedure maximizes the sensitivity of the instrument in the mass region of interest for the PAH analysis.

9. Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis an aliquot of internal standard solution is transferred to the sample vial using a 250 uL syringe to give a final internal standard concentration of 40 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following, or similar conditions:

Injector Temp - 250°C
Transfer Line Temp - 290°C
Initial Oven Temp - 30°C
Initial Hold Time - 1 min.
Ramp Rate - 10°C/min.
Final Temperature - 325°C

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each PAH as shown in Table 1. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

10. Calculations

10.1 Qualitative Identification

Obtain EICPs for the primary m/z and the confirmatory ion. The following criteria must be met to make a qualitative identification:

The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

For the qualitative identification, the relative retention time (RRT) of unknown peaks fall within +/- 0.06 RRT units.

The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. In some instances a compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive data includes correct relative retention time and the presents of the secondary ion but the ratio is greater than ± 20% of the primary ion which may be caused by an interference of the secondary ion. When the primary ion is not affected by interferences and the decision is agreed to by the reviewer, the compound is flagged with an asterisk (*) on the sample summary sheet.

Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.2 Quantitation

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = (A_S C_{is})/(A_{is} C_S)$$

where:

A_S = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
C_{is} = Concentration of the internal standard, (ng/mL).
C_S = Concentration of the parameter to be measured, (ng/mL).

Based on these response factors, sample extract concentrations for each PAH is calculated using the following formula.

$$Ce = \frac{(A_S)(I_S)}{(A_{is})(RF)}$$

where:

Ce = Sample extract concentration (ng/mL)
A_S = Area of the characteristic ion for the parameter to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
I_S = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = (Ce) \times \frac{V_E}{V_S},$$

C = Concentration in Sample (ng/L)
V_E = The final extract volume (mL), and
V_S = The original volume of sample extracted (L).
Ce = The ng/mL amount measured in the analytical extract.

11. Quality Control/Quality Assurance

11.1 GC/MS Tuning

The GC/MS is tuned as described in section 8.0.

11.2 GC/MS Initial Calibration and Continuing Calibration Check

Prior to the use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

Each calibration standard is analyzed and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound at each concentration level is calculated using the following equation:

$$RF = \frac{A_s}{A_{is}} \times \frac{C_{is}}{C_s}$$

A_s = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the internal standard

C_s = Concentration of the compound to be measured.

For every 12 hours of GC/MS analysis, the mass spectrometer response (RF) for each PAH of interest (Table 1) relative to the internal standard is determined.

These daily response factors for each compound must be compared to the initial calibration curve. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{\overline{RFI} - RFC}{\overline{RFI}} \times 100$$

$\overline{RFI}$ = Average response factor from initial calibration.

RFC = Response factor from current verification check standard.

If the daily response factor are within ±35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ±35 percent of the corresponding calibration curve value, a five- point calibration curve must be repeated for that compound prior to the analysis of samples.

11.3 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

Method blank analysis are performed at the rate of one per case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

If the method blank contains any of the carcinogenic PAHs listed in Table 10-3 at concentrations greater than the method detection limit (MDL), or any other target PAH compound at a concentration 5 times greater than the MDL, the method blank will be considered out of control. Corrective action will include reanalysis of the blank extract, an investigation into laboratory sources of contamination and qualifying that sample data relates to the blank. Blank level contamination should be considered the minimum level of contamination in all samples that are analyzed with the blanks.

* A case is a group or a set of samples collected from a particular site over a given period of time.

11.4 Surrogate Compound Analysis

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and will measure individual sample matrix effects associated with sample preparation and analysis. Surrogates will include naphthalene-d_8, fluorene-d_{10}, and chrysene-d_{12}.

RMAL will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

Surrogate	Acceptance Criteria % Low-Level
Naphthalene-d8	21-108
Fluorene-d10	41-162
Chrysene-d12	10-118

The following corrective action will be taken when required as stated above:

a) Check calculations to assure there are no errors;

b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;

c) If the surrogate recovery is outside the control limits, the secondary ion may be used to check the quantitation of the surrogate. If the secondary ion meets within the control limits this recovery is reported with flag of # next to the percent recovery.

d) If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that an interference specific to the surrogate was present that resulted in high recovery and this interference would not affect the quantitation of other target compounds. The presence of this type of interference can be confirmed by evaluating the chromatographic peak shapes in ion intensities of the surrogate.

e) If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as D with the note surrogate diluted out.

f) Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis of the extract yields surrogate recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

11.5 Matrix Spike Analysis

The laboratory will spike and analyze 5% matrix spike samples. RMAL will spike seven representative compounds into water. These compounds and the spiking levels as listed in section 4.6. The initial matrix spike criteria for data validity are as follows:

SPIKE COMPONENT	ACCEPTANCE CRITERIA
1H-INDENE	20-150
NAPHTHALENE	20-150
QUINOLINE	20-150
2-METHYLNAPHTHALENE	20-150
FLUORENE	69-118
CHRYSENE	20-132
BENZO(E)PYRENE	20-150

One compound is allowed to be below the above acceptance criteria. The average recovery for the spike pair must also fall into the above criteria with one compound being allowed below the acceptance criteria.

Criteria for data validity for each individual matrix spike compound will be developed as data is collected and will be updated annually.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, then the reanalysis data will be used. If not, the data for the sample will be reported but qualified as being outside the acceptance criteria of the method. Both the original and reanalysis data will be reported.

11.6 Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound. Corrective action will be performed if the relative difference is greater than 70% for target compounds.

TABLE 1

COMPOUNDS AND MS QUANTITATION MASS IONS*

Compound	Quantitation Mass Ion	Confirmation Ion	Internal Standard Reference
Polynuclear Aromatic Hydrocarbons (PAH)			
Naphthalene	128	102	1
Acenaphthylene	152	151	1
Acenaphthene	154	153	1
Fluorene	166	165	1
Phenanthrene	178	176	2
Anthracene	178	176	2
Fluoranthene	202	200	2
Pyrene	202	200	2
Benzo(a)anthracene	228	226	3
Chrysene	228	226	3
Benzofluoranthenes	252	250	3
Benzo(a)pyrene	252	250	3
Indeno(1,2,3,cd)pyrene	276	274	3
Dibenz(a,h)anthracene	278	279	3
Benzo(g,h,i)perylene	276	274	3
Internal Standards			
1) Acenaphthene-d10	164		--
2) Phenanthrene-d10	188		--
3) Benzo(a)pyrene-d12	264		--

* The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICP's must fall within +/- 20% of the relative intensities of these masses in a reference mass spectrum.

TABLE 1 (Continued)

Compound	Quantitation Mass Ion	Confirmation Ion	Internal Standard Reference
Surrogates			
1) Naphthalene-d8	136	134	1
2) Fluorene-d10	176	174	1
3) Chrysene-d12	240	236	3
Heterocycles and Other PAH			
Indene	116	115	1
Indole	117	90	1
2,3-dihydroindene	117	118	1
2,3-benzofuran	118	90	1
Quinoline	129	102	1
Benzo(b)thiophene	134	89	1
2-methylnaphthalene	141	115	1
1-methylnaphthalene	141	115	1
Biphenyl	154	153	1
Carbazole	167	166	2
Dibenzofuran	168	139	1
Acridine	179	178	2
Dibenzothiophene	184	139	2
Perylene	252	250	3
Benzo(e)pyrene	252	250	3
7,12-Dimethylbenz(a)anthracene	256	241	3
3-Methylcholanthrene	268	252	3

TABLE 2

RELATIVE RETENTION TIMES AND CONFIDENCE FOR THE COMPOUNDS ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

	Absolute Retention Time (minutes)	Avg. RRT	SD	% RSD	95% Confidence Limits
Benzofuran	8:03	0.550	0.015	2.807	0.520-0.580
Dihydroindene	8:45	0.590	0.016	2.765	0.558-0.622
Indene	8:54	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8(Surr.)	11:14	0.733	0.017	2.289	0.699-0.767
Naphthalene	11:16	0.735	0.017	2.289	0.701-0.769
Benzo(b)thiophene	11:25	0.743	0.017	2.258	0.709-0.777
Quinoline	12:06	0.783	0.017	2.140	0.749-0.817
Indole	12:55	0.824	0.018	2.167	0.788-0.860
2-methylnaphthalene	12:59	0.832	0.017	2.084	0.798-0.866
1-methylnaphthalene	13:15	0.848	0.017	2.055	0.814-0.882
Biphenyl	14:12	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	15:15	0.962	0.018	1.822	0.927-0.988
Acenaphthene	15:44	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	16:09	1.011	0.018	1.791	0.975-1.047
Fluorene-d10(Surr.)	16:57	0.872	0.015	1.735	0.842-0.902
Fluorene	17:01	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	19:08	0.974	0.016	1.617	0.942-1.006
Phenanthrene	19:28	0.988	0.016	1.589	0.956-1.020
Anthracene	19:34	0.994	0.016	1.597	0.962-1.026
Acridine	19:42	0.999	0.016	1.572	0.967-1.031
Carbazole	20:02	1.013	0.015	1.487	0.983-1.043
Fluoranthene	22:32	1.130	0.017	1.461	1.096-1.164
Pyrene	23:07	1.157	0.017	1.443	1.123-1.191
Benz(a)anthracene	26:16	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	26:18	0.874	0.012	1.320	0.850-0.898
Chrysene	26:22	0.876	0.012	1.320	0.852-0.900
Benzofluoranthenes	29:00	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	29:34	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	29:44	0.988	0.016	1.615	0.956-1.020
Perylene	29:55	0.996	0.016	1.644	0.964-1.028
Indeno(1,2,3 cd)pyrene	32:31	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	32:36	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	33:17	1.149	0.028	2.422	1.093-1.205

STANDARD
OPERATING
PROCEDURE

Subject or Title: Total Recoverable Phenolics - City of St. Louis Park (Manual)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1112	1.0	June 13, 1990

Supersedes: Original

1. Scope and Application

   1.1 This method measures steam-distillable phenolic materials which react with the color reagents under the conditions of the analysis.

   1.2 The detection limit is 5 ug/L as Phenol.

   1.3 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.

   1.4 The range extends to 0.1 mg/L. The range can be extended by dilution of the samples.

   1.5 Approximate preparation time is 2 hours for a group of 10 samples. Analytical time is about 15 minutes per sample.

2. Summary of Method

   The sample is acidified and distilled to separate phenolics from interfering compounds. Phenolics in the distillate react with 4-aminoantipyrene in the presence of potassium ferricyanide at pH 10 to form a reddish-brown dye, which is extracted into chloroform and measured colorimetrically at 460 nm.

3. Comments

   3.1 Interferences

      3.1.1 Most direct interferences are eliminated by distillation of an acidified sample. Phenolic compounds distill with the water but interfering compounds do not.

Prepared by:	Date:
Lindsay Breyer	June 13, 1990
Management Approval: Anne Lang	Date: June 14, 1990
QA Officer Approval: John Zimmerman	Date: June 15, 1990

3.1.2 Some phenolic compounds are not steam-distillable and will not be determined.

3.1.3 The colors produced by various phenolic compounds are not the same, so the response will depend on the compounds actually present in the samples. Phenol has been selected as the calibration standard since it is not possible to reproduce the mixture of compounds present in the sample. The result obtained will represent the minimum concentration of phenolics present in the sample.

3.1.4 Interference from sulfur compounds is eliminated by acidification and addition of copper sulfate.

3.1.5 Oxidizing agents such as chlorine will oxidize phenolic compounds and must be removed.

3.1.6 Oil may distill over and interfere with the analysis.

3.1.7 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 Wear gloves, apron, and face shield when performing distillations. Distillations are to be performed under the slot hood.

4.4 Phenol is extremely toxic and can be absorbed through the skin. Handle only in a fume hood and wear gloves. In case of skin contact, flush with water for at least 15 minutes. Notify your supervisor or safety officer of any exposures.

4.5 Extractions are to be performed in a fume hood. Wear gloves and apron. Take care to keep chloroform vapors confined to the hood.

4.6 Samples, reagents and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

5. Samples Collection and Preservation

5.1 Samples are to be collected in glass containers and preserved by adding sulfuric acid to pH < 2 and refrigerating at 4°C.

5.2 The holding time is 28 days.

6. Apparatus

6.1 All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller, and associated adapters and hardware.

6.2 Recirculating chiller.

6.3 pH meter and electrode.

6.4 Separatory funnels, 500 mL, with supporting rack.

6.5 Porcelain spot-test plate.

6.6 Spectrophotometer with 2 cm cells.

6.7 Filter funnels.

6.8 Filter paper, Whatman 41.

6.9 Micropipettes with disposable tips, 10 uL, 20 uL, 1 mL.

6.10 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Sulfuric Acid, 50%

Slowly add 500 mL concentrated sulfuric acid to 500 mL deionized water with constant mixing and cool. The reaction is very exothermic and should be done with extreme caution.

7.2 Boiling stones.

7.3 Copper Sulfate, 10%

Dissolve 100 g cupric sulfate 5-hydrate in deionized water and dilute to 1000 mL.

7.4 Ferrous Ammonium Sulfate Solution

Add 1 mL concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.5 Buffer Solution

Dissolve 16.9 g ammonium chloride in 143 mL concentrated ammonium hydroxide and dilute to 250 mL with deionized water. Prepare this solution in a hood. Two milliliters of this solution should adjust the pH of 100 mL distillate to 10.

7.6 Aminoantipyrene Solution

Dissolve 2.0 g of 4-aminoantipyrene in deionized water and dilute to 100 mL.

7.7 Potassium Ferricyanide Solution

Dissolve 8 g potassium ferricyanide in deionized water and dilute to 100 mL.

7.8 Phenol Stock Standard, 1000 mg/L

Dissolve 1.000 g phenol in deionized water and dilute to 1000 mL.

7.9 Phenol Intermediate Standard, 1.0 mg/L

Dilute 1.0 mL 1000 mg/L Stock Standard to 1000 mL with deionized water.

7.10 Working Standards

Dilute the 1.0 mg/L Intermediate Standard with deionized water as follows:

Aliquot (mL)	Final Vol. (mL)	Conc. (mg/L)
0	200	Blank
1.0	200	0.005
2.0	200	0.010
4.0	200	0.020
10.0	200	0.050
20.0	200	0.100

Note: The standards are not distilled with the samples.

7.11 pH test strips

7.12 Starch/iodide test strips

7.13 Lead Acetate test strips

8. Procedure

8.1 Sample Preparation

8.1.1 Measure and record the pH of all water samples. pH test strips may be used.

8.1.2 Check for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet.

8.1.3 Check for sulfide using lead acetate test strips. A dark color indicates the presence of sulfide. Record the result on the bench sheet.

8.1.4 Measure 200 mL sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 2.0 g and add 200 mL deionized water. Be sure to adjust the pH of soil and waste samples before distillation. Record the exact weight on the bench sheet.

8.1.5 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

8.1.6 If the pH is not < 2, add 50% sulfuric acid dropwise until it is.

8.1.7 If the sulfide test was positive, add 2 mL 10% copper sulfate.

8.1.8 Assemble the distillation apparatus, turn on the cooling water and hood, and start the distillation. Capture the distillate in a 250 mL beaker.

8.1.9 When 150 to 175 mL distillate has been collected, turn off the heating mantle and allow to cool.

8.1.10 Add 25 to 30 mL deionized water and resume distillation until 200 mL has been collected. Turn off the heating mantle and clean out the flask when cool. Do not over distill the samples as this will lead to interferences in the analysis.

8.1.11 Transfer the distillates to 250 mL glass bottles with teflon caps and refrigerate until the are analyzed.

8.2 Spot Test

8.2.1 Place 1 mL aliquots of each sample in the wells of a porcelain spot test plate. Also run a blank (deionized water) and the 0.10 mg/L standard.

8.2.2 Add 20 uL buffer solution to each well and stir.

8.2.3 Add 10 uL aminoantipyrene solution and stir.

8.2.4 Add 10 uL potassium ferricyanide solution and stir.

8.2.5 Compare the color of the samples to the color of the blank and standard. Any samples appearing darker than the standard will require dilution prior to analysis. Make note of these on the bench sheet along with the estimated dilution required. If necessary, dilute the sample and spot check the dilution.

8.3 Dilution Technique

8.3.1 Since the sample volumes may not be exactly 200 mL after distillation, it is not possible to make dilutions volumetrically. Dilutions must be done on a weight basis.

8.3.2 Place a beaker on a top loading balance and zero it.

8.3.3 Pour the entire sample into the beaker and note the weight.

8.3.4 Divide the weight by the required dilution factor to determine the sample weight to be analyzed. For example, if there are 205 g distillate and a 10x dilution is needed, 20.5 g of the distillate should be analyzed.

8.3.5 Measure out this weight of sample for analysis and dilute to a total volume of 200 mL. Return the unused portion of the sample to the original container. Record all dilutions made on the bench sheet.

8.4 Analysis of Samples

8.4.1 Place 200 mL sample (or standard) in a 500 mL separatory funnel. Analysis should be performed in a hood.

8.4.2 Add 4 mL buffer solution and mix.

8.4.3 Check the pH with a pH meter (pH paper is not sensitive enough). The pH should be 10 $\pm$ 0.2. If necessary, adjust the pH by dropwise addition of ammonium hydroxide or hydrochloric acid.

8.4.4 Add 2 mL aminoantipyrene solution and mix.

8.4.5 Add 2 mL potassium ferricyanide and mix.

8.4.6 Wait 3 minutes, then add 25 mL chloroform.

8.4.7 Shake the separatory funnel 10 times. Vent chloroform fumes into the hood. Then allow the phases to separate.

8.4.8 Shake the funnel another 10 times and let the chloroform settle.

8.4.9 Filter the chloroform extracts through filter paper into 2 cm cuvettes.

8.4.10 Measure and record the absorbances at 460 nm, zeroing on chloroform; not the blank.

8.4.11 If any samples measure higher than the highest standard, up to a 5x dilution may be made on the extract with chloroform. Record the dilution on the bench sheet and make it clear that the chloroform extract was diluted, as the calculation will be affected.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (deionized water) is required with every batch of 20 less samples. The blank must be taken through the entire prep and analysis with the samples. Additional blanks, termed "Initial Calibration blank" (ICB) and "Continuing Calibration Blank" (CCB) are also analyzed. These blanks are used only to evaluate the determinative step and are not distilled. They are analyzed at a frequency of one ICB per 20 samples and one CCB per 10 samples.

9.1.2 The calibration is verified by the analysis of two different laboratory check standards. An "Initial Calibration Verification" (ICV) check standard is analyzed at a frequency of one per 20 samples. This check is carried through the entire procedure, including the distillation step. The measured value from this check standard must be between 75% and 125% of the true value.

A "Continuing Calibration Verification" (CCV) check standard is analyzed at a frequency of one per 10 samples. This standard is used to verify the determinative step only. The measured value must be between 85% and 115% of the true value.

If the measured values from the check standards are not within control limits, the system is out of control and corrective action must be performed.

9.1.3 Save the original blank and standards; new ones do not have to be extracted.

9.1.4 Duplicate analyses are performed at a frequency of 5%. Corrective action is performed if the relative difference from the duplicate analysis is greater than 70%.

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \ Project: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

SOW No. 7/88

RMA Sample No. Client Sample ID.

\
\
\
\
\
\
\
\
\

\
\
\
\
\
\
\
\
\

PARAMETERS	METHOD	DETECTION LIMIT	SOURCE
Phenolics	420.1	5 ug/L	1

COMMENTS:

\
\

SOURCE:
1="Methods for Chemical Analysis of Water and Wastes," USEPA-EMSL, Cincinnati.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

1
INORGANIC ANALYSIS DATA SHEET

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \

Matrix (soil/water): \ SDG No.: \

Level (low/medium): \

% Solids: \

RMA Sample No.
\
Client Sample ID.
\

Project: \

Date Received: \

Concentration Units (ug/L or mg/kg dry weight): \

Analyte	Concentration	C	Q	M
Phenolics				

Color Before: \ Clarity Before: \ Texture: \

Color After: \ Clarity After: \ Artifacts: \

Comments:

\
\

2A
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No. \ SDG No.: \

Initial Calibration Source: \

Continuing Calibration Source: \

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					M
	True	Found	%R(1)	True	Found	%R (1)	Found	%R (1)	
Phenolics									

3
BLANKS

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Preparation Blank Matrix (soil/water): \

Preparation Blank Concentration Units (ug/L or mg/kg): \

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prepa-ration Blank	C	M
Phenolics											

5A
SPIKE SAMPLE RECOVERY

RMA Sample No.
\
Client Sample ID.
\

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Matrix: \ Level (low/medium): \

% Solids for Sample: \

Concentration Units (ug/L or mg/kg dry weight): \

Analyte	Control Limit % R	Spiked Sample Result (SSA)	C	Sample Result (SA)	C	Spike Added (SA)	% R	Q	M
Phenolics									

Comments: \

6
DUPLICATES

RMA Sample No. \
Client Sample ID. \

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Matrix (soil/water): \ Level (low/medium): \

% Solids for Sample: \ % Solids for Duplicate: \

Concentration Units (ug/L or mg/kg dry weight): \

Analyte	Control Limit	Sample (S)	C	Duplicate (D	C	RPD	Q	M
Phenolics								

FORM VI - IN

7/88

HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

Introduction

This Health and Safety Plan applies to personnel who will potentially be exposed to groundwater affected by creosote or coal tar constituents during the retrieval of groundwater samples from active pumping wells, the GAC plant, monitor wells, and piezometers. This plan has been designated to comply with, as a minimum, the requirements set forth in 29 CFR 1910.120, the OSHA standards governing hazardous waste operations. In no case may work be performed in a manner that conflicts with the intent of or the safety concerns expressed in this plan.

Materials of Concern and Effects of Overexposure

The materials of concern which have been identified for this project are coal tar and creosote related materials including naphthalene, other polynuclear aromatic hydrocarbons (PAH) and phenolic compounds.

Coal tar and creosote are typically irritating to the eyes, skin and respiratory tract. Acute skin contact may cause burning and itching while prolonged contact and poor hygiene practices may produce dermatitis. Prolonged skin contact with creosote must be avoided to prevent the possibility of skin absorption.

Naphthalene is a hemolytic agent which, upon overexposure to the vapor or ingestion of the solid, may produce a variety of symptoms associated with the breakdown of red blood cells. Naphthalene is also irritating to the eyes and repeated or prolonged contact has been associated with the production of cataracts.

Repeated exposure to certain PAH compounds has been associated with the production of cancer. Contact of PAH compounds with the skin may cause photosensitization of the skin producing skin burns after subsequent exposure to ultraviolet radiation.

Phenolics are generally strong irritants which can have a corrosive effect on the skin and can also rapidly penetrate the skin. Overexposure to phenols and phenolic compounds may cause convulsions as well as liver and kidney damage.

Hazard Assessment

Initial

Because of the relatively low vapor pressures associated with PAH compounds (generally less than 10^{-4} mm Hg at $20^{o}C$), they are not expected to present a vapor hazard. The most likely threat of exposure to these compounds will be via skin contact.

TABLE 1

ACTION LIMITS FOR AIR CONTAMINANTS

Limit	Persistent Concentration in the Breathing Zone	Procedure
Lower	5 ppm	Don respirators, step up monitoring.
Upper	50 ppm	Stop work and back off from immediate work area until levels subside in the breathing zone.

Action Limits

The American Conference of Governmental Industrial Hygienists (ACGIH) has established threshold limit values (TLV) for phenol and naphthalene at 5 and 10 ppm, respectively, as 8-hour time weighted averages (TWA). Based on these values, the action limits in Table 1 have been set. The lower limit of 5 ppm is based on the TLV for phenol while the upper limit of 50 ppm is based on a minimum protection factor of 10 for a half-mask, air purifying respirator.

Response

When the PID yields persistent breathing-zone readings at or above the lower action limit, workers in the affected area will don respirators. Air sampling will continue on a more frequent basis. If readings are persistent at or above the upper limit, workers shall back off from the immediate work area until measured breathing-zone concentrations fall below the lower limit, at which time operations will resume and normal air monitoring will continue. If breathing zone levels do not fall below the upper limit, workers are to leave the work area and report the condition immediately to the City, the Engineer, or its representative. If necessary, engineering controls will be instituted to maintain vapor concentrations below the upper limit or arrangements will be made to upgrade to Level B protection.

Personal Protective Equipment

Personal protective equipment (PPE) will be donned, as necessary, based on the hazards encountered. Listed below is the personal protective equipment to be utilized during this project and the conditions requiring its use.

Personal Protective Equipment

- Coveralls - Polyethylene coated Tyvek if work involves contact with affected soil or groundwater.
- Boots - Chemical resistant type if work involves contact with affected soil or groundwater.
- Hard Hat - When working in the vicinity of operating heavy machinery.
- Face shield - If splash hazard exists.
- Gloves - Nitrile for potential contact with affected soil or groundwater.
- Respirator - MSA Comfo II with GMC-H Cartridges if PID reading exceeds 5 ppm or if dust or odors become objectionable.
- Chemical Safety Goggles - If eye irritation occurs.

Because of the carcinogenicity of certain PAH compounds, and because of the skin hazards associated with PAH and phenolic compounds, it is important that appropriate protective clothing be worn during work activities, which may involve the possibility of skin contact with affected soil or groundwater. As a minimum, the presence of visible creosote or coal tar related material shall constitute evidence of affected soil or groundwater.

Health and Safety Training

Personnel covered by this Health and Safety Plan must have received appropriate health and safety training prior to their working on the site. Training will include:

- Requirements for and use of respirators and personal protective equipment.
- Required personal hygiene practices.
- Requirements for employees to work in pairs.
- Proper material handling.
- Proper sampling procedures.
- Maintenance of safety equipment.
- Effective response to any emergency.
- Emergency procedures.
- Hazard zones.
- Decontamination methods.
- General safety precautions.

A copy of the Standard Safety Procedures (Table 2) will be given to each worker covered by this Health and Safety Plan.

Decontamination

Administrative procedures require hygienic practices consistent with work hazards. Employees will be instructed in the training program on proper personal hygiene procedures.

Contaminated, reuseable PPE, such as boots, hard hats, face shields and goggles, will be decontaminated prior to leaving the site. The decontamination procedure follows:

- Rinse with water to remove gross contamination.
- Wash in Alconox or equivalent detergent solution.
- Rinse with clean water.

Contaminated, disposable PPE, such as Tyvek coveralls and gloves will be placed in 55-gallon drums and stored while arrangements are made for disposal.

TABLE 2

STANDARD SAFETY PROCEDURES

- Employees are required to work in pairs.
- Wash face and hands prior to eating, smoking, or leaving the site.
- No smoking or eating is allowed in the work area during excavation or sampling activities.
- Wearing of contact lenses is not permitted in the work area.
- Contaminated material (e.g., Tyvek coveralls) must be properly disposed of before leaving the site.
- All work must be conducted in accordance with local, state and federal EPA and OSHA regulations, particularly 29 CFR 1910.120.

Respirators, if used, will be cleaned and disinfected after each day of use. The facepiece (with cartridge removed) will be washed in a hypochlorite (or equivalent) disinfecting solution, rinsed in warm water and air dried in a clean place.

Emergency Procedures

This Health and Safety Plan has been established to allow site operations to be conducted without adverse impacts on worker health and safety as well as public health and safety. In addition, supplementary emergency response procedures have been developed to cover extraordinary conditions at the site.

General

All accidents and unusual events will be dealt with in a manner to minimize a continued health risk to site workers. In the event that an accident or other unusual event occurs, the following procedure will be followed:

- First aid or other appropriate initial action will be administered by those closest to the accident/event. This assistance will be conducted so that those rendering assistance are not placed in a situation of unacceptable risk. In the event that a worker is caught in a trench collapse, call for emergency assistance immediately.

- All accidents/unusual events must be immediately reported to the Owner.

- All workers on site should conduct themselves in a mature, calm manner in the event of an accident/unusual event, to avoid spreading the danger to themselves, surrounding workers and the community.

Responses to Specific Situations

Emergency procedures for specific situations are given in the following paragraphs.

Worker Injury

If an employee in an affected area is physically injured, Red Cross first-aid procedures will be followed. Depending on the severity of the injury, emergency medical response may be sought.

If the injury to the worker is chemical in nature (e.g., overexposure), the following first-aid procedures are to be instituted:

- Eye Exposure - If affected solids or liquids get into the eyes, wash eyes immediately using large amounts of water and lifting the lower and upper lid occasionally. Obtain medical attention immediately.

- Skin Exposure - If affected solids or liquids get on the skin, promplty wash the affected skin using soap or mild detergent and water. Obtain medical attention immediately when exposed to concentrated solids or liquids.

- Inhalation - If a person inhales large amounts of a toxic vapor, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Obtain medical attention as soon as possible.

- Swallowing - When affected solids or liquids have been swallowed, the Poison Control Center will be contacted and their recommended procedures followed.

Emergency Notification

In an extraordinary event that might be damaging to personnel or adjacent property, immediate notification of the proper emergency service will be required. The proper emergency service is determined by the nature of the emergency.

EMERGENCY NOTIFICATION

Fire Department	911
Ambulance	911
Police Department	911
Methodist Hospital	932-5000
Poison Control Center	347-3141

OTHER CONTACTS

MPCA - Douglas Beckwith	612-296-7715
EPA - Darryl Owens	312-886-7089
City of St. Louis Park - James Grube	612-924-2551
- William Gregg	612-924-0117

COMMUNITY RELATIONS PLAN

COMMUNITY RELATIONS PLAN

The Sampling Plan is to be completed in accordance with the Consent Decree-Remedial Action Plan for Reilly Tar & Chemical Corporation's St. Louis Park, Minnesota, N.P.L. Site. All community relations programs related to this work will be coordinated through the following agencies:

United States	**Ms. Judy Beck** United States Environmental Protection Agency (312) 353-1325
State of Minnesota	**Mr. Ralph Pribble** Minnesota Pollution Control Agency (612) 296-7792
City of St. Louis Park	**Mr. James N. Grube** City of St. Louis Park (612) 924-2551

Information necessary to conduct the Community Relations Plan will be provided by the City and Reilly.